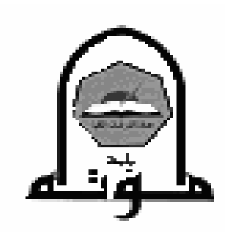


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Deanship of Graduate Studies

Determination of Trace Metal Concentrations in Contaminated Soil of Karak Solid Waste Disposal Site, Jordan

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This Thesis was submitted in Partial Fulfillment of the Requirements for the Master's Degree in Chemistry
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الآراء الواردة في الرسالة الجامعية لا تُعبر
بالضرورة عن وجهة نظر جامعة مؤتة



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استكمالاً لمتطلبات الحصول على درجة الماجستير في **الكيمياء**.

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DEDICATIONS

To my Father who was my first teacher……………………………
To my Mother who encourages me through all my life……………
To my wife and my son…………………………………………….
To my brothers and sister ………………………………………….
To my friends and each person who gave help, encouragement and assistance to let this thesis be a success.

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First and foremost, I should offer my thanks, obedience and gratitude to "ALLAH" who helped me and gave me the strength to achieve my thesis.

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Mohammad Naji Al Tarawneh

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Abbreviations

EC	Electrical conductivity
EF	Enrichment factor
CRM	Certified reference material
DTPA	Diethylene triaminepentaacitic acid
TEA	Triethanol amine
GIS	Geographical Information Systems
GPS	Global Positioning System
MAPEP	Mixed-Analyte Performance Evaluation Program
FAAS	Flame Atomic Absorption Spectrometer
ppt	Parts per trillion
ppm	Parts per million
TOC	Total organic carbon
LOD	Limits of detection
LOQ	Limit of quantitation
RSD	Relative standard deviation
R^2	Correlation coefficients
USDA	US Department of Agriculture
PFA	Perfluoroalkoxy resin
SD	Standard deviation
AAS	Atomic Absorption Spectrometer

Abstract

Determination of Trace Metal Concentrations in Contaminated Soil of Karak Solid Waste Disposal Site, Jordan

Mohammad Naji Al Tarawneh

Mu'tah University, 2012

The present study was aimed to determine the concentration of trace metals and their distribution pattern in top soil of new Karak Solid Waste Disposal site. Additionally, the work was planned to optimize a microwave digestion method using acid mixture and validate the analytical method using certified reference material for determination of trace metal concentrations using atomic absorption spectrometry (AAS). Seventy five soil samples were collected during autumn 2011 from the investigated solid waste disposal site located 25 km north-east of Karak City.

The soil samples were analyzed for bulk physio-chemical parameters such as pH, Total Organic Carbon (TOC%), Electrical Conductivity (EC), Calcium Carbonate percentage ($CaCO_3$%) and soil texture. The concentration of trace metals (Zn, Ni, Pb, Cu, Co, Cr, Fe and Mn) were analyzed in collected soil samples in total and available forms in order to identify the distribution pattern in the investigated area and to evaluate their mobility and uptake. The pH values of investigated samples were ranged from 7.86 to 8.97 with an average of 8.26, TOC% between 0.91 to 3.26% with an average of 2.19%, EC between 1.3 to 24.6 mS/cm with average of 9.94 mS/cm and the soil texture was characterized as sandy loam to loamy sand. The total concentration of the elements Zn, Ni, Pb, Cu, Co, Cr, Fe and Mn were ranged between 45.51-589.65, 17.42 - 327.76, 55.88 - 369.35, 31.83-213.85, 86.87-180.90, 52.94-533.74, 267.02-831.85 and 91.26-241.40 mg/kg, respectively. Furthermore, the ratio of available heavy metals to total concentration were as follows Zn: 4.28%, Ni: 1.43%, Pb: 1.87%, Cu: 0.96%, Co: 0.63%, Cr: 0.71%, Fe: 0.39% and Mn: 6.70%. The Enrichment factor calculations showed that Pb and Co were highly enriched compared to other elements and the order of enrichment factor was found as follows; Pb >> Co > Ni > Cr > Cu > Zn > Mn. The relative availability ratio of studied metals showed a high Mn availability and consequently the metals mobility in the surface soils followed the order of Mn>Zn>Pb> Ni> Cr> Cu> Co> Fe. While, Fe was found strongly retained in the soil.

الملخص

تحديد تراكيز بعض العناصر الثقيلة وتوزيعها على سطوح التربة في مكب النفايات الصلبة الجديد في محافظة الكرك

محمد ناجي الطراونه

جامعة مؤتة، 2012

هدفت الدراسه الحالية لتحديد تركيز العناصر الثقيلة وتوزيعها على سطوح التربة في مكب النفايات الصلبة الجديد الواقع في محافظة الكرك ، حيث تم القيام بتحديد افضل طريقة لهضم العينات بإستخدام جهاز المايكروويف وخليط من الاحماض ، كما تم كذلك فحص طريقة التحليل ومدى فاعلتها باستخدام المواد المرجعية لتحديد تركيز العناصر الثقيلة بإستخدام جهاز طيف الإمتصاص الذري. لقد تم جمع 75 عينة من التربة في مكب النفايات الصلبة المشار الية والواقع على بعد 25 كم شمال شرق مدينة الكرك خلال خريف عام 2011 .

حيث تم حساب العديد من الخواص الفيزيائية والكيميائية للعينات مثل درجة الحموضه، الايصالية الكهربائية، المادة العضوية، نسبة كربونات الكالسيوم والنسجة للتربة. كذلك تم حساب التركيز الكلي والمتيسر لكل من العناصرالتاليه (الزنك، النيكل، الرصاص، النحاس، الكوبالت، الكروم، الحديد والمنغنيز) وتم التعرف على طريقة توزع هذه العناصر في منطقة الدراسة وتقدير حركتها ومقدار استيعاب التربه لتراكيزها. وقد بلغت درجة حموضة العينات من (7.8 - 8.97) بمعدل 8.26 و المادة العضوية من (0.91% - 3.26%) بمعدل 2.19% وتراوحت قيمة الايصالية الكهربائية بين(1.3 - 24.6) ملي سمنز\سم بمعدل 9.94 ملي سمنز\سم لجميع العينات وتميز نوع النسجة بالتربه الرملية المتوسطة. وتراوح التركيز الكلي للعناصر الثقيلة من (45.5 - 589.65)، (17.42 - 327.76)، (55.88 - 369.35)، (31.83 - 213.85)، (86.87 - 180.)، (52.94 - 533.74)، (267.02 - 831.85)، (91.26 - 241.40) ملغم\كغم لكل من العناصر التالية الزنك، النيكل، الرصاص، النحاس، الكوبالت، الكروم، الحديد والمنغنيز على التوالي. وبلغت نسبة التركيز المتيسر للعناصر إلى التركيز الكلي بمقدار .28% ، .43% ، .87% ، .96% ، .63% ، 0.71%، .39% ، 6.70% لكل من الزنك، النيكل، الرصاص، النحاس، الكوبالت، الكروم، الحديد والمنغنيزعلى التوالي. وأخذت العينات الترتيب الاتي عند إحتساب عامل الاثراء (الرصاص > الكوبالت > النيكل > الكروم > النحاس > الزنك > المنغنيز) . وقد اثبتت الدراسة ان المنغنيزهو الاكثر تيسرا وحركه في التربة السطحيه من بين العناصر الاخرى كما هو موضحا بالترتيب الاتي (المنغنيز>>الزنك>الرصاص>النيكل >الكروم>النحاس>الكوبالت >الحديد)، ومن جهه اخرى وجد أن الحديد هو العنصر الاكثر ثباتا والذي يبقى لفترات أطول داخل قطاع التربه.

Chapter one
Theoretical Background

1.1 Conception of waste disposal

The solid waste disposal means to put waste into a landfill for the purpose of final burial destruction or placement for future recovery. As the terminal action for pollution control of solid waste, the final target of disposal is isolating solid waste and its environmental impact from biosphere and to keep humankind and environment from any unacceptable hazard from the infection of hazardous components in wastes. The objective of final disposal is that the wastes cannot be processed and used further (Yuan, 2001).

1.2 Solid waste in Jordan

The main sources of solid waste generation in Jordan can be attributed to the following :

a. Municipal residential waste.
b. Commercial waste.
c. Residual waste from waste water treatment plants.
d. Industrial waste.
e. Institutional waste.
f. Agricultural waste .

The average waste generation rate in Jordan was estimated in the range of 0.34 to 1.07 kg/Capita/day with an average of 0.91 kg/Capita/day (Chopra et al. 2001). The estimated solid waste generation rates in different countries compared to Jordan are shown in Table 1.1. It was observed that the solid waste generation rate in Jordan was higher than the rate for developing countries and lower than developed countries (Chopra et al. 2001). Furthermore, the composition of solid waste in Jordan was consisted mainly of organic matter (50 – 68%), papers (5-10%), metals (3-6%), glass (2-5%), and plastics (4-6%), as shown in table 1.2. However, the composition of solid waste in Al Karak governate was estimated as Kitchen residues (52%), plastics (5%), papers (4%), agriculture residues (15%), glass (3%), metals (4%), textile (15%) and others (2%) (SWDF, 2011), Figure 1.1.

Table (1.1)

Comparison of solid waste generation rates in Jordan with other countries (Chopra et al. 2001)

Location	Per Capita Generation Rates (g $cap^{-1}day^{-1}$)
Jordan	910
Bangalore, India	400
Manila, Philippines	400
Asuncion, Paraguay	460
Seoul, Korea	2000
Vienna, Austria	1180
Mexico City, Mexico	680
Paris, France	1430
Australia	1870
Sunnyvale, California, USA	2000

Table (1.2)

Comparison of the Jordan Solid Waste contents with other Countries (GCEP. 2001)

	The percentage per weight			
Waste Kind	Jordan	Asian Countries	UK	USA
Organics	50-68	75	3.6	20
Papers	5-10	2	31.2	43
Metals	3-6	0.1	5.3	7
Glass	2-5	0.2	3.8	9
Plastics	4-6	0.1	5.2	5

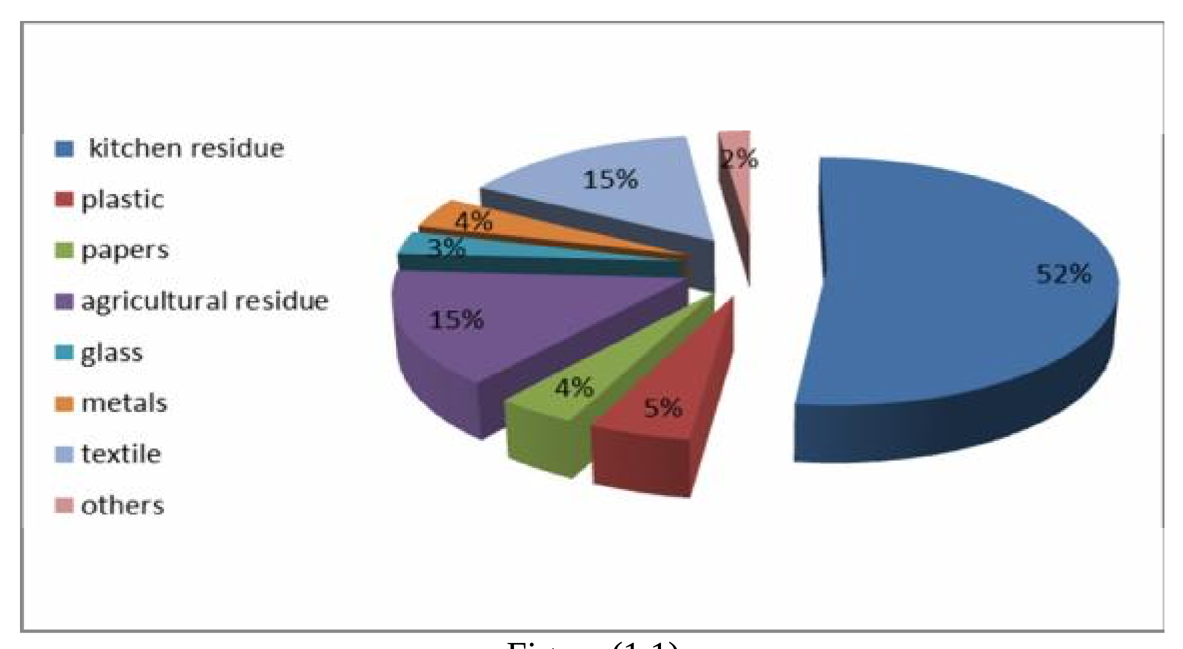


Figure (1.1)

Composition of solid waste disposed at Alkarak disposal site (SWDF, 2011)

1.3 Heavy Metals

The trace and heavy metals are terms usually interchangeable used to describe a group of metallic element (Harte et al, 1991). The trace metals are metals that present either in the environment or in the human body in very low concentrations such as Cu, Fe, and Zn. While, heavy metals such as Cd, Pb, and Hg are those trace metals whose densities are at least five times greater than water density. Additionally, some of these metals may be called toxic metals like Pb and Cd whose concentration in the environment are considered to be harmful to biota.

In soil, the metal or contaminant becomes environmentally toxic depending on several factors namely: (i) prevailing soil physical and chemical condition such as acidity and presence of clays, Fe-Mn oxides and soil organic matter which provide adsorption and reaction surfaces, (ii) soil and site hydrology which cannot only dilute the contaminant in situ effect but can also transport the pollution effect from its source to cause contamination in new unaffected sites and (iii) the plant and soil microbial components of ecosystem which uptake and recycle metals (Ross, 1996).

Much research focuses on understanding the effect of metals on food and plant production. Less interest has been focused on issues of trace metals cycling in natural ecosystem (Ross, 1996). A simplified illustration of the storage compartment and fluxes involved in the cycling of metals in a soil-plant system is given in Figure (1.2).

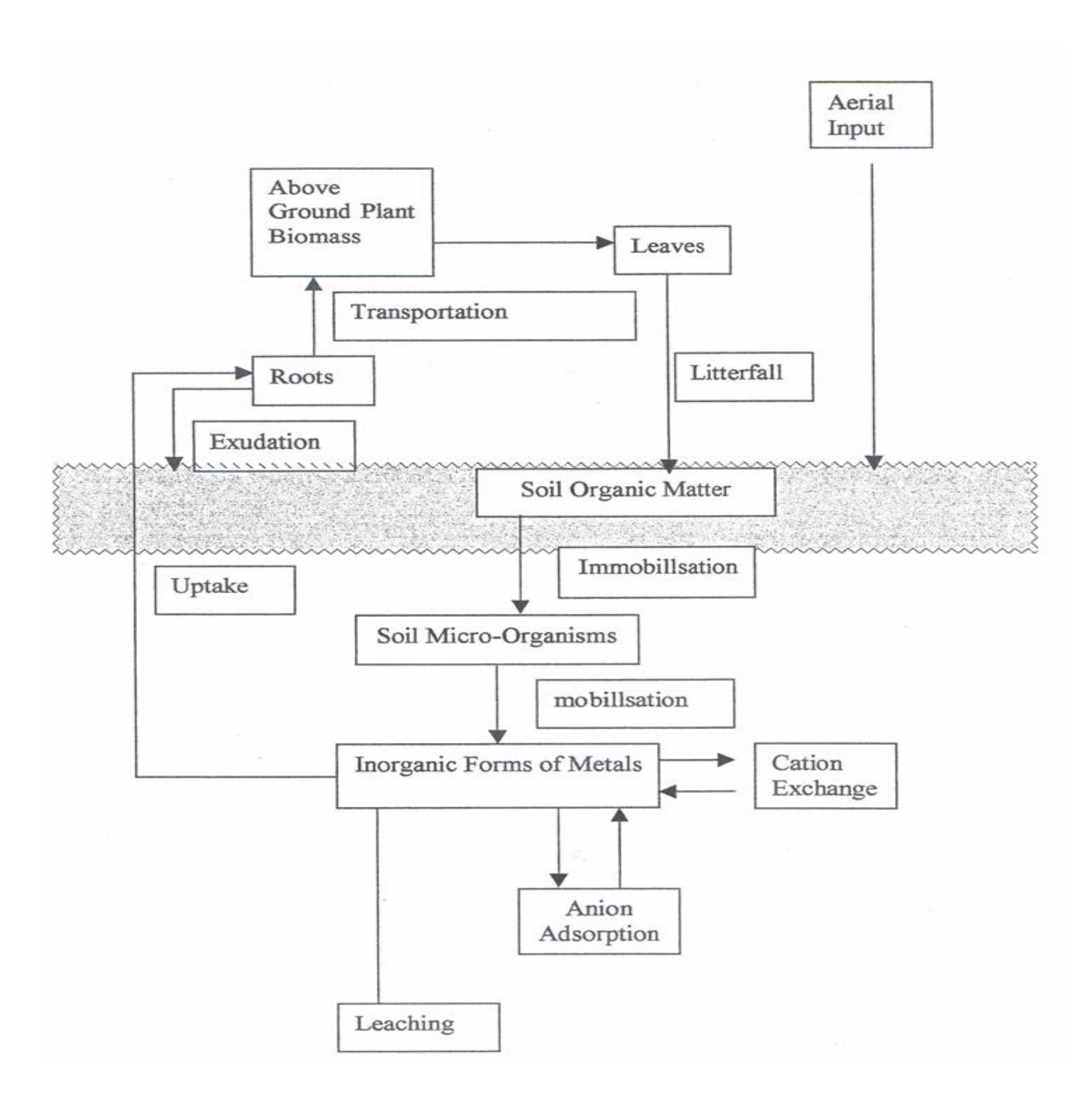


Figure (1.2)
Storage compartment and fluxes involved in the cycling of metals in soil-plant system. (Ross, 1996)

1.4 Principal sources of heavy metals

Mineral rock weathering and anthropogenic activities provide two of the main types of metal input to soil-plant system. Higher quantities of certain metals are being released into the environment by anthropogenic activities, primary associated with industrial processes, manufacturing, and disposal of industrial and domestic waste materials. Five main groups of anthropogenic sources of trace metals contamination in soil are listed in table (1.3) (Ross, 1996).

Table (1.3)
Sources of Toxic Metals in the Environment

1- Metalliferous mining and smelting:

a. Spoil heaps and tailings-contamination through weathering, wind erosion (As, Cd, Hg, and Pb)
b. Fluvially dispersed tailings-deposited on soil during flooding, river dredging, etc.(As, Cd, Hg, and Pb)
c. Transported ore separates-blown from conveyance onto soil (As, Cd, Hg, and Pb)
d. Smelting-contamination due to wind-blown dust, aerosols from stack (As, Cd, Hg, Pb, Sb, and Se)
e. Iron and steel industry (Cu, Ni, and Pb)
f. Metal finishing (Zn, Cu, Ni, Cr, and Cd)

2. Industry

a. Plastics (Co, Cr, Cd, Hg)
b. Textiles (Zn. Al,Ti, Sn)
c. Microelectronics (Cu, Ni, Cd, Zn. Sb)
d. Wood Preserving (Cu, Cr, As)
e. Refineries (Pb, Ni, Cr)

3. Atmospheric Deposition:

a. Urban/industrial sources, including incineration plants, refuse disposal (Cd, Cu, Pb, Sn, Hg, and V)
b. Pyrometallurgical industries (As, Cd, Cr, Cu, Mn, Ni, Pb, Sb, Tl, Zn)
c. Automobile exhausts (Mo, Pb, (with Br and Cl), V)
d. Fossil fuel combustion (including power stations) (As, Pb, Sb, Se, V, Zn, Cd)

4. Agriculture:

a. Manures (e.g. As, and Cu in some pig and poultry manures, Mn and Zn in farmyard manure)
b. Lime (As, Pb)
c. Pesticides (Cu, Mn, and Zn in fungicides, As and Pb used in orchards)
d. Irrigation waters (Cd, Pb, and Se)
e. Corrosion of metals (e.g. galvanized and metal objects (fencing, troughs, etc.)(Fe, Pb, and Zn)

5. Waste disposal on land:

a. Sewage sludge (Cd, Cr, Cu, Hg, Mn, Mo, Ni, Pb, V, and Zn)
b. Leachate from landfill (As, Cd, Fe, and Pb)
c. Scrap heaps (Cd, Cr, Cu, Pb, and Zn)
d. Bonfires coal ash. etc. (Cu, and Pb)

1.5 Heavy Metal Features and Health

Heavy metal toxicity is frequently the result of long-term, low level exposure to common pollutants in our environment: air, water, food and numerous consumer products. Exposure to toxic heavy metals is associated with many chronic diseases and can cause a wide variety of health problems.

Table (1.4) summarizes exposure source and health effects of some heavy metals (Harte et al,1991;Moore et al., 1984 and Brady,1982).

Table (1.4)

Exposure sources of some heavy metals and healthy effects.

Element	Cadmium Cd
Exposure Source	Air pollution, paints, welding, seafood, cigarette smoke, coffee & tea, nickel cadmium batteries, pipe smoke, fungicides, pesticides, contaminated water, some plastics.
Symptoms	Fatigue, headaches, high blood pressure, kidney disorders, skin disorders, learning disabilities, increase risk for cancer, loss of smell.
Element	**Lead (Pb)**
Exposure Source	Paints, delivery and manufacture of petroleum products, smoke exposure, eating/cocking in ceramic glazed dishware, leaded gasoline, living inter-city, canned foods, eating contaminated liver, cigarette smoke, news print and colored ads, hair dyes and rinses, rubber toys, storage batteries, cosmetics, pottery.
Symptoms	Fatigue, high blood pressure, learning disabilities. targets heart, brain liver, bone. Poor bone growth anxiety, muscle and joint pain, anemia, weakness, target liver bone, ... etc.
Element	**Zinc (Zn)**
Exposure source	Food, meat, fish, dairy products, cereal and grain eating and drinking foods stored or cooked in galvanized metal containers.
Symptoms	Increase the risk of heart disease, rapid breathing, shivering, fever, sweating chest.
Element	**Copper (Cu)**
Exposure source	Pesticides, water, use of Cupper vessels for cooking or surveying vessel, hot water passing copper pipes.
Symptoms	Reduce the stability of vitamin A&C; reduce the ability of enzyme in human.
Element	**Manganese (Mn)**
Exposure source	Welding, pesticides, steel factories, mining dust, burnt gasoline (when gasoline is used as additive), underground water ... etc.
Symptoms	Risk of health problem like nervous system.
Element	**Iron (Fe)**
Exposure source	Welding, steel factories, heavy traffic, mining dust, Fertilizers industries and coloring, most of mechanical maintenance works.
Symptoms	Increase of iron can cause fibrosis of the pancreas, diabetes, heart failure . . .etc.

1.6 Literature Review

Many studies had been conducted to investigate the heavy metals contamination in soil worldwide. In general, it was observed that there was a significant increase in the total content of the heavy metals in soil closer to the

pollution source, which clearly decreases with increasing the distance. The soil might be contaminated with heavy metals such as Cd, Zn Pb, Mn, Fe, Al, Cu and Ni due to industry, agriculture, metalliferous mining ,smelting and waste disposal on land. Industrial waste and effluents are being discharged randomly in soils or in the vicinity of industrial operations, usually contain high amounts of heavy metals such as Cd, Fe, Mn, Pb, Zn, Ni, Cr, and Cu. (Abul Kashem et al., 1999; Shallari et al., 1998; Fang et al., 2000; Collet et al., 1998; Meneses et al., 1999). In the vicinity of the industries around Dhaka city in Bangladesh, the concentration of total Cd, Cu, Mn, Ni, Pb and Zn ranged from 0.1-1.8, 28-217, 106-577, 25-112, 17-99 and 53-477 mg/ kg soil, and respectively, they found that among the industrial sites the concentration decreases with increasing distance from the disposal points (Monacci et al. 1997). In the industrial site of Bangladesh, the maximum concentrations of Cd, Co, Cr, Cu Ni, Pb and Zn were 14, 476, 3865, 1107, 3579, 172, and 2495 mg/kg, respectively (Abul Kashem et al. 1999).

Many trace metals are present in leaded and unleaded petrol, diesel oil, anti-wear substances added to lubricants, brake pads, and tires, and emitted by vehicle exhaust pipes. Lead is added to gasoline to prevent engine knock, so that the cars become a major source of lead to the environment (Monacci et al 1997). Recently leaded gasoline was banned in several countries worldwide. Metals such as Fe, Cu and Zn are essential components of many alloys, pipes, wires and tires in motor vehicles and are released into the roadside environment as a result of mechanical abrasion. Heavy metal content of roadside soil was measured as a function of both distance from highway and the depth (ward et al., 1975,Yassoglou et al., 1987, and Jaradat et al., 1999). The results showed that contamination decreases exponentially with increasing the distance from the road edge and dropped to the background level at about 50- 100m (Ward et al., 1975;Yassoglou et al., 1987, and Jaradat et al., 1999).It was also shown that most of the lead was concentrated in the upper 5 cm of soil (Yassoglou et al., 1987). The level of heavy metal contamination was found to increase with traffic volume (Nicholas et al, 1990; Ho et al., 1988, and Jaradat et al., 1999). Heavy metals are released in varying quantities into agriculture soil from phosphatic fertilizer and pesticide. (Harte et al., 1991).

The heavy metal concentration in agriculture soils has been investigated in many studies . The mean concentration of heavy metals in agriculture soil in Korea was found as: 0.118-0.146 (Cd), 4.938-6.078 (Pb), and 0.045-0.11 (Hg), 2.823-3.501(Cu), 4.694-7.818 (Zn) and 0.355-0.782 (As) mg/kg on dry-weight basis (Kim et at, 1999). Significantly higher content of Pb, Cd, Cu, Zn were found in the soil of the urban area compared with those of suburban

ones, where traffic proved to be an important source of soil pollution in the urban areas (wong et al., 2002;poon et al., 2001; Mellor et al., 1999 and (sanka et al., 1995). Many studies investigated the heavy metal concentration profiles in the soil (Merry et al., 1983). The results showed that very high concentrations were found in the top few centimeters of the soil and then the heavy metal concentration decreases with depth and reaches the background levels between 30 and 100 cm. The concentrations of heavy metals Pb, Cd, Cu, Zn, Cr, and Fe in street dust and in soil in near the Jordanian Petroleum refinery were studied (Momani et al., 2002).The results showed that the level of heavy metals in street dust is higher than in the soil sample due to automobiles and refinery emission products.

Elevated levels of Cu, Pb, Cd, and Zn were found in roadside soil compared with the background values. It was found that at 1.5m east of the highway, the average concentrations were 29.7,0.75,188.8, and 121.7,mg/kg for Cu Cd Pb and Zn, respectively (Jaradat et al., 1999).

1.7 Goal of Study

The current study was intended to determine the trace metals distribution in top soil of new Karak Soild Waste Disposal site. The work is planned to optimize a microwave digestion method and validation of analytical method for determination of trace metal concentrations using atomic absorption spectrometry (AAS).

Specific goals:

1. Validation of analytical procedure for the determination of metal in semiarid to arid soil in Jordan using acid microwave digestion and atomic absorption spectrometry (AAS).
2. Investigation of trace metals in soil affected by solid waste disposal site.
3. Studying the distribution pattern of trace metals in the investigated area.

Chapter Two
Experimental

2.1 Study area

The study area is the solid Waste Disposal site which is located around 25 km north-east of Karak City, Figure (2.1). The site has an area around 40 Acre and located between 31^{o} 220'N to 31^{o} 230'N latitude and 35^{o}. 935'E to 35^{o}. 950'E longitudes. The municipal solid waste site is an active disposal site which collects the solid waste from Karak City and its property since 1996. Overall it can be stated that current landfill operations are causing environmental degradation and adverse effects on surrounding areas.

The solid waste site is receiving an estimated amount around 700-800 ton/day with an average composition of 52% kitchen residue, 5% plastics, 4% papers, 15% agricultural residue, 3% glass, 4%, metals, 15%textile, and 2% others, (SWDF, 2011). The solid waste is collected from Karak area and transported with trucks to the disposal site on daily basis.

The study area is classified as an arid to semiarid region. It has an average rainfall quantity less than 200 mm/annually which occurs mainly during the rainy season that extends from December to March and the rest of the year is warm and dry. The mean Annual temperature is 18^{0}C (SWDF, 2011). The average daily wind speed is 20-30 km/h and the direction of wind blowing mainly from east to southeast (SWDF, 2011). Sampling activities had taken place during the autumn of 2011 to avoid rain-washing out of the heavy metal.

2.2. Sample collection

The sampling area was divided as an array of 15 column and 5-7 rows as shown in Figure 2.1. The Global Positioning System (GPS) coupled with Geographical Information Systems (GIS) were used to locate points in the field. The GPS was used to establish the boundaries of the study area, then use steel stakes to locate sampling quadrates. In total seventy five (75) soils composite samples were collected from the solid waste disposal site. They were labeled serially (1-75); each sample represents a composite of three sub-samples from each sampling site. The samples were taken from the top layer (0-15 cm) using a stainless steel trowel as illustrated in figure (2.1). Six control soil samples were collected from areas located far away from the dumping site and not affected by dumping solid waste. they were designated as reference site.

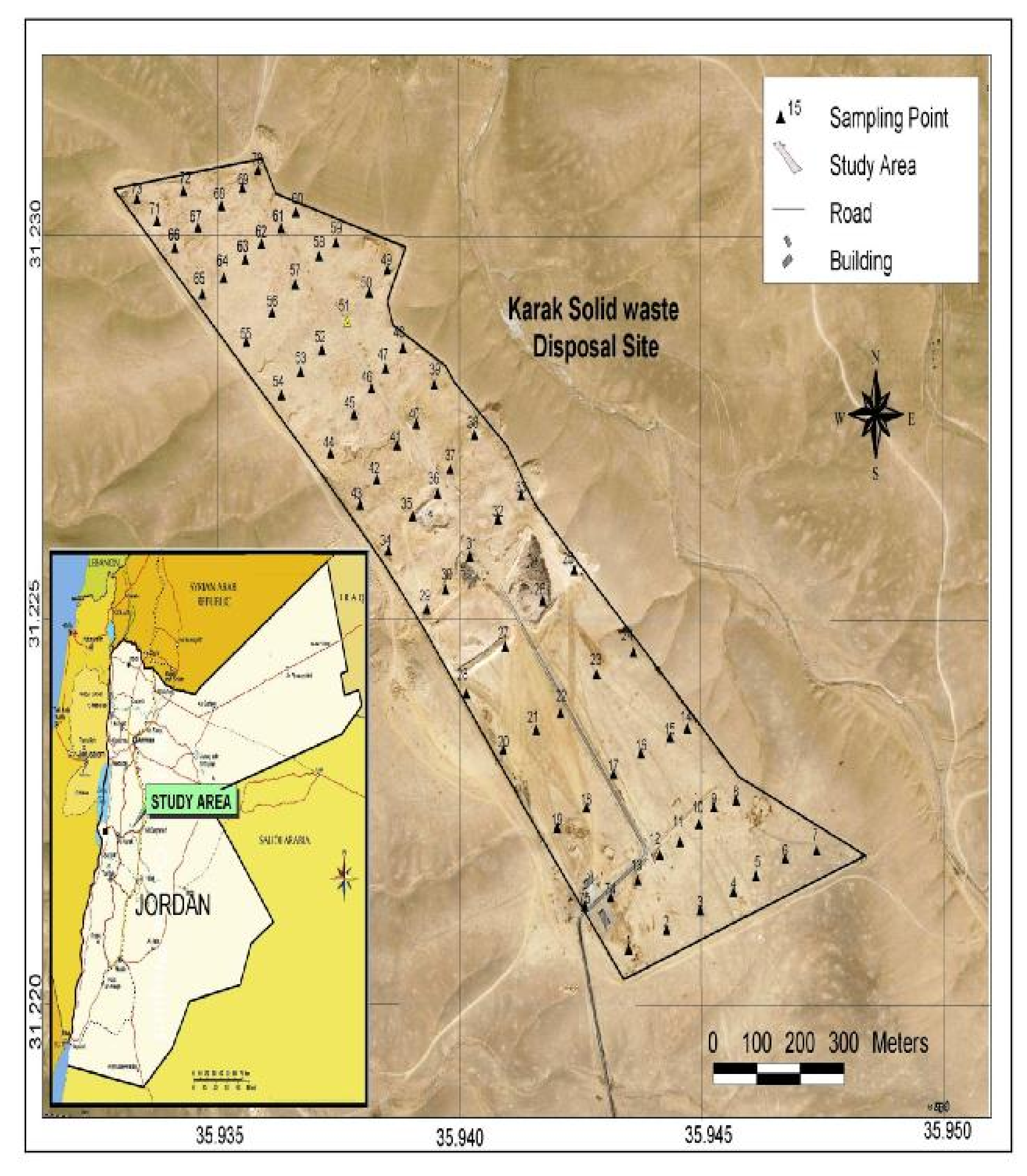


Figure (2.1)
GIS location map of sampling sites of karak solid waste disposal site and exact longitudes and latitudes of the sampling sites.

2.3 Sample characterization

2.3.1 Sample pre-treatment

To assure sample stability and homogeneity, the collected soil samples were air dried, grounded using a ceramic mortar with pestle, then they were sieved through 75µm (mesh 200) stainless steel sieve and stored refrigerated in pre-washed plastic bags for subsequent sample preparation, microwave digestion and analysis.

2.3.2. pH and Electrical Conductivity (EC)

The soil pH values were determined by shaking 1:2.5 (w/w) ratio of soil and deionized water, the mixture was homogenized for 6 h using horizontal shaker before the measurement with the pH-meter model 525 (WTW®, Weilheim, Germany) (Abul Kashem et al.,1999). Electrical conductivity was measured in a saturated extract of soil using an EC-meter model LF-320 (WTW®, Weilheim, Germany))

2.3.3. Calcium carbonate content

The calcium carbonate content ($CaCO_3$) of eight samples was determined by rapid titration method of Piper as reported in (Hess, 1971).

2.3.4. Total organic carbon (TOC %)

The total organic carbon (TOC %) was determined using the wet oxidation method according to Walkley-Black´s method (Baruah and Bathakur, 1997). To 0.5-1.0 g of oven dried and fine- grained soil, 10 mL of 1 M $K_2Cr_2O_7$ and 20 mL of concentrated sulfuric acid (H_2SO_4) were added. The mixture was gently shaken and allowed to stand for 30 min until completion of the oxidation reduction reaction. The mixture was diluted with 200 mL of distilled water, and then 10 mL of 85 % phosphoric acid (H_3PO_4) and 1.0 mL of diphenylamine indicator were added. Finally, the mixture was back titrated against 0.5M ferrous ammonium sulfate ($Fe(NH_4)_2(SO_4)_2.6H_2O$), until violet color was observed. A blank titration was carried out in the similar way using all reagents except the soil, and then TOC % was calculated according to the following formula:

$$TOC\ \% = (B-S) \times 0.5 \times 0.003 \times (100/W) \times 1.3 \times 1.724$$

Where:

B: volume in mL of ferrous ammonium sulfate needed for blank titration,

S: volume in mL of ferrous ammonium sulfate needed for sample titration,

W: weight in grams of sample, 0.5 is the molarity of ferrous ammonium sulfate, 0.003 is grams of organic carbon, 1.3 is Walkey correction factor and 1.724 is von Bemmlen factor.

2.3.5 Soil texture analysis

Soil texture was determined using the hydrometer method (Baruah and Bathakur, 1997). A 50 g of oven-dry soil was taken and treated with 60 mL of 6 % H_2O_2 in order to remove the organic matter; and then it was heated over water bath at 80-90 ^oC. The process was repeated until frothing disappeared. The residual content was transferred to a 800 mL beaker and diluted with 400 mL distilled water, then 100 mL of Calgon reagent (5% of sodium hexametaphosphate) was added. The suspension was stirred using magnetic stirrer for 15-20 min, then it was transferred into settling cylinder (1 L) which was sealed with Para-film and shaken for 1 min. then the hydrometer was inserted immediately into the settling cylinder and after 4 min, hydrometer reading (a) and suspension temperature were recorded. Finally, reading of hydrometer (b) after 2 hours and the suspension temperature were recorded. The hydrometer readings were corrected according to its calibration temperature. The particles size distributions were determined as follows:

Silt % = (a-b)/c×100

Clay % = (b/c) ×100

Sand % = 100-(Silt % + Clay %)

Where:

a: the corrected hydrometer reading after 4 min.

b: the corrected hydrometer reading after 2 hours.

c: is the weight in gram of the oven-dry sediment sample after subtraction of the weight of the oxidized organic matter.

2.4. Sample preparation

2.4.1. Total Heavy Metal Digestion

Soil samples were digested using an Anton Parr microwave digestion system model Multiwave 3000 (Anton Parr®, Österreich). Accurately weighing 0.30 g of ground soil sample which was then transferred to pre cleaned PFA Teflon vessels and suitable amounts of acid mixture were added according to the optimization procedure of the microwave digestion, table 2.1. The digestion PFT vessels were capped and placed inside the rotor body, sealed, tightened and submitted to microwave digestion programs. The reference samples were treated in the same manner as the samples. Each series of digestion included a sample blank which was a pre-cleaned Teflon vessel containing the same type and amount of the reagents but without the soil

sample. Several digestion procedures were tried to achieve complete decomposition, clear solutions and the best recovery rate. After microwave digestion, the digested samples were quantitatively transferred to polypropylene bottle and diluted to a final volume
of 50 mL. They were kept in the refrigerator until analysis.

Table (2.1)
Microwave program of four digestion methods using the CRM soil sample.

Procedure Method	Sample size	Reagent (ml)		Ramping Time (min)	Temp ^0C	Pressure Bar	Power (watt)
A	0.3 g	HNO_3 H_2O_2 HF	7 ml 2ml 4ml	5	210	40	Up to 800
B	0.3g	HNO_3 HCl HF	6ml 2ml 2ml	5	210	40	Up to 800
C	0.3 g	HNO_3 HCl	3ml 7ml	5	210	40	Up to 800
D	0.3g	HNO_3 HF	7ml 3ml	5	210	40	Up to 800

2.4.2. Bioavailability Heavy Metal Extraction

The Bioavailability of Heavy Metals was determined by the extraction with Diethylene Triamine Pentaacetic acid (DTPA) (Lindsay and Norvell, 1978). The DTPA extraction method was used for determination of the bioavailability of heavy metals in soil samples. The extracting solution was consisted of 0.005M DTPA, 0.01M calcium chloride and 0.1M TEA (triethanol amine) at pH 7.3.Accurately weighing 10.00 g of air-dried soil into a 125ml polyethylene erlenmeyer flask. Then 50ml of DTPA solution was added and the flask caped tightly with a polyethylene stopper. It was shaken using a horizontal shaker for 2h, then the mixture was decanted and filtered through filter paper into 50ml polyethylene bottle. Then it was acidified with four drops of concentrated nitric acid (HNO_3), and the heavy metals concentrations were determined using Atomic Absorption Spectrometer (AAS).

2.5. Method development and optimization

2.5.1. Method validation

Several digestion procedures were performed to achieve a complete decomposition, clear solutions and the best recovery rate. The certified reference material (CRM) (MAPEP-07-MaS18) purchased from Mixed-

Analyte Performance Evaluation Program (MAPEP, USA) as digested using four different acid mixtures in the microwave and recovery rates were determined for each method. The digestion methods are summarized in Table (2.1). The results of the recovery rate showed that procedure B gave the best result and it was used to digest soil, reference and blank samples.

2.5.2. Quality control

A quality control protocol including reagent blanks, replicate samples, and certified reference material (CRM) were used to assess the precision and the accuracy of analytical procedure. Blanks were prepared in a similar manner to that of soil samples and were analyzed before each measurement. All analysis were made with replicate samples (n=3) for quality control assessment and the average results were reported. External calibration method was used to build up calibration curves for each element using different concentration levels, the calibration curve was accepted according to USEPA which exceeds ($r^2 > 0.995$) (Ming and Lena ,1998).

2.5.2.1 Limit of detection (LOD)

The limit of detection (LOD) is the smallest concentration or amount of an analyte that can be reliably shown to be present or measured under defined conditions (Christian, 2004). The LOD for any analytical procedure, the point at which analysis is just feasible, may be determined by a statistical method based on measuring replicate blank samples. The limit of detection is the lowest concentration level that can be determined to be statistically different from an analyte blank. There are numerous ways that detection limits have been defined, LOD was calculated according to the following formula:-

$$\mathbf{LOD = 3.3(s/S)}$$

Where:

s:- standard deviation for blank sample.

S:- the slope or sensitivity of the calibration curve for the heavy metals.

2.5.2.2 Limit of Quantitation (LOQ)

The limit of quantitation (LOQ), or concentration at which quantitative results can be reported with a high degree of confidence (Christian, 2004), may likewise be determined by a statistical method or by an empirical method too.

The empirical method of LOQ is based on signal to noise ratio (S/N 10:1) for standard solutions. However, the LOQ can be determined to be statistically different from an analyte blank as follows:-

$$LOQ = 10(s/S)$$

Where

s: - standard deviation for blank sample.

S: - the slope or sensitivity of the calibration curve for the heavy metals.

The presence of a substance may still be detected qualitatively but not quantitatively with sufficient degree of reliability. In case, the presence of a substance that cannot be detected with sufficient reliability, the content is below the detection limit, and the substance is considered as non detectable

2.6. Chemical and Instrumentation

2.6.1. Glassware

All glassware and polyethylene bottles were initially cleaned with soap, washed thoroughly with tap water , distilled water, and socked in 1%HNO_3 (v/v) for overnight. To remove any contamination by heavy metals,

2.6.2. Chemicals and Materials

Chemicals and materials were used for sample preparation, digestion, quantification of heavy metals, clean up, titration, and other analytical procedures were of analytical grade. They are listed in Table 2.2.

Table (2.2)

Chemical materials and reagents.

Chemical substance	Abbreviation	Company
Nitric acid (70%)	HNO_3	Sigma-Aldrich/ USA
Hydrochloric acid (38%)	HCl	Sigma-Aldrich/ USA
Hydrofluoric acid (40%)	HF	Sigma-Aldrich/ USA
Hydrogen peroxide (35%)	H_2O_2	Sigma-Aldrich/ USA
Sulfuric acid	H_2SO_4	Sigma-Aldrich/ USA
Ferrous ammonium sulfate hexahydrate (99%)	$(Fe(NH_4)_2(SO_4)_2 . 6H_2O)$	Panreac/Espana
Potassium dichromate (99.5%)	$K_2Cr_2O_7$	Ridial/Germany
Phosphoric acid (85%)	H_3PO_4	Koch Light Research Laboratories Pty Ltd
calcium chloride	$CaCl_2$	Ridial/Germany
Diphenyl amine indicator	For TOC	Sigma-Aldrich/ USA
diethylene tri amine penta acitic acid	DTPA	GCC/England
tri ethanol amine	TEA	GCC/England
(1000ppm)Standard Solution		Dionex/California

*All reagents used in extraction and quantification procedures were analytical grade.

2.6.3. Instrumentation

2.6.3.1 Microwave digestion system

The samples were digested using a microwave digestion system model Multiwave 3000 (Anton Parr®, Österreich). With 100-mL PFA Teflon vessels (MF 100) and 16-position. Rotor. The conditions for the microwave digestion system were summarized in Table 2.1.

2.6.3.2 Atomic Absorption Spectrometer (AAS)

Heavy Metals concentrations were measured using an atomic absorption spectrometer (AAS) Model AA-6200 (Shimadzu®, Kyoto, Japan). The AAs was equipped with a hollow cathode lamp and a 10 cm long slot-burner head and air/acetylene flame. The operating conditions adjusted in the spectrometer were carried out according to the standard guidelines of the manufacture. The hollow cathode lamp and emission wavelength, slit width, the correct coefficient for the calibration straight line, the working linear range and detection limit found for each metal.

The conditions of Atomic Absorption Spectrometer are presented in Table 2.3.

Table (2.3)

Standard conditions used in determination of different elements using atomic absorption Spectrometer. (Shimadzu, AA-6200**)**

Element	Wave length (nm)	Slit width (nm)	Flow of fuel (L/min)	Lamp current (mA)	Flame description
Pb	217.0	0.7	2.0	12	Air-Acetylene
Cu	324.7	0.7	1.8	6	Air-Acetylene
Fe	248.3	0.2	2.2	12	Air-Acetylene
Mn	280.1	0.2	2.0	10	Air-Acetylene
Zn	213.9	0.7	2.0	8	Air-Acetylene
Cr	357.9	0.7	2.8	10	Air-Acetylene
Co	240.7	0.2	2.2	12	Air-Acetylene
Ni	232.0	0.2	2.2	12	Air-Acetylene

2.6.3.3 pH and EC meters

The pH was analyzed using glass electrode pH meter model pH 525 (WTW®, Weilheim, Germany). The pH meter was calibrated based on pH 4 and pH 7 buffer solutions at 25^{o}C according to standard method (Andrew et al., 2005). In addition EC was measured by conductivity meter model LF-320 (WTW®, Weilheim, Germany). The EC-meter was calibrated using 0.01 M KI at 25^{o}C according to standard method (Andrew et al., 2005).

2.6.3.4 Geographic information system (GIS)

The coordination (longitudes, latitude) of the study area were cited using the geographic information system (GIS) apparatus model (GPS II plus®, USA)

2.6.3.5 Other equipment's

Other laboratory equipment's were used such as oven (J.P.SELECTA®, Spain), horizontal shaker (KS 501 digital®, Germany), sieves (CISA,RP.09®, Spain) ,and high precision analytical balance with five decimals(AND-HR-202®, Japan).

Chapter Three
Results and Discussion

3.1. Physical and chemical soil properties

Some physical and chemical soil properties were determined for all collected samples including: pH, EC, Total Organic Carbon (TOC), Calcium Carbonate Content ($CaCO_3$ %), and soil texture. Additionally, the total and available heavy metals content were determined in the investigated area to evaluate the pollution load and its effect on the element concentration levels and distribution pattern. The following sections are presenting the results and discussing these parameters in details in addition to the validation of the analytical method.

3.1.1. pH

The results of pH measurements showed that all soil samples were moderately basic, as shown in table 3.1. The pH values of soil in the investigated area were ranged from 7.86 to 8.97 with an average of 8.26, which was higher than pH value in the control area (7.62), Figure 3.1.The minimum pH value was found at site 40 (7.86) and the maximum at site 2 (8.97). By comparing pH range values at the investigated and control sites, it was noticed that higher variation in pH values at investigated area rather than the control area which can be explained due to difference in waste composition that might affect the pH value such as the kitchen/food residues. Additionally, the high pH value in the investigated area can be attributed due to the high percent of carbonate content (Lasat, 2000; Ge et al., 2000), which caused the neutralization of soil acidity (Momani, 2003).This means that the changes of soil acidity can influence neutral compositions in the soils by removing the bivalent base (like $Ca^{2+}$and Mg^{2+}) from soil (Kim et al,2003) . It is known that alkaline soil prevent the metals mobility and lead to their precipitation in the soil (McCauley et al, 2003.)

Table (3.1)
Physical and chemical soil parameters

Sample	TOC%	pH	EC*	Sample	TOC%	pH	EC
1	1.95	8.36	5.70	24	0.94	8.55	2.80
2	2.35	8.97	7.20	25	1.31	8.23	2.20
3	2.02	8.10	12.30	26	1.98	8.03	1.60
4	3.03	8.35	9.90	27	2.02	8.31	3.60
5	2.69	8.15	10.40	28	1.68	8.32	6.20
6	3.19	8.08	12.90	29	1.51	8.39	2.70
7	1.58	8.35	5.60	30	2.35	8.47	1.40
8	1.21	8.30	7.40	31	2.15	8.22	9.70
9	1.82	8.29	8.30	32	2.12	7.99	18.90
10	2.15	8.32	8.50	33	2.42	8.09	12.30
11	2.52	8.44	6.30	34	1.48	8.37	14.90
12	2.29	8.34	8.30	35	1.45	8.08	9.60
13	2.08	8.26	8.90	36	2.96	8.33	9.00
14	0.91	8.27	3.60	37	3.26	8.37	7.90
15	2.49	8.23	6.20	38	2.49	8.40	8.30
16	2.42	8.09	2.70	39	1.38	8.15	9.70
17	2.22	8.27	1.40	40	2.02	7.86	8.90
18	2.35	8.22	9.70	41	1.34	8.02	13.70
19	2.69	8.35	18.90	42	2.55	8.19	11.80
20	2.02	8.55	12.30	43	2.02	8.37	16.40
21	1.51	7.96	14.90	44	1.65	8.06	13.20
22	2.89	8.22	9.60	45	2.35	8.23	10.30
23	0.67	8.41	9.00	46	2.08	8.52	9.40

Continue Table 3.1

Sample	TOC%	pH	EC*	Sample	TOC%	pH	EC
47	2.55	8.43	2.10	65	2.35	8.07	10.80
48	2.79	8.83	3.80	66	2.49	8.01	17.30
49	2.25	8.18	1.30	67	1.34	8.6	25.20
50	2.42	8.45	15.10	68	2.35	8.38	2.20
51	2.69	8.30	13.20	69	1.98	8.08	15.70
52	2.02	8.18	7.10	70	2.22	8.24	20.10
53	1.01	7.93	9.70	71	2.15	8.19	16.60
54	2.29	8.15	8.80	72	2.55	8.13	16.30
55	2.59	8.49	8.30	73	2.72	7.90	17.30
56	3.06	8.09	14.50	74	2.89	8.26	7.40
57	2.86	7.93	24.60	75	3.19	8.19	4.00
58	2.49	8.51	17.30	Control 1	0.67	7.20	4.10
59	2.69	8.07	19.70	Control 2	1.01	7.40	2.70
60	3.26	8.18	14.20	Control 3	0.94	7.80	3.60
61	2.82	8.57	21.10	Control 4	1.04	8.01	3.90
62	1.68	8.27	13.40	Control 5	0.64	7.51	2.98
63	2.02	8.08	8.90	Control 6	0.77	7.82	3.1
64	1.82	8.17	6.00				

*EC :(mS/cm)

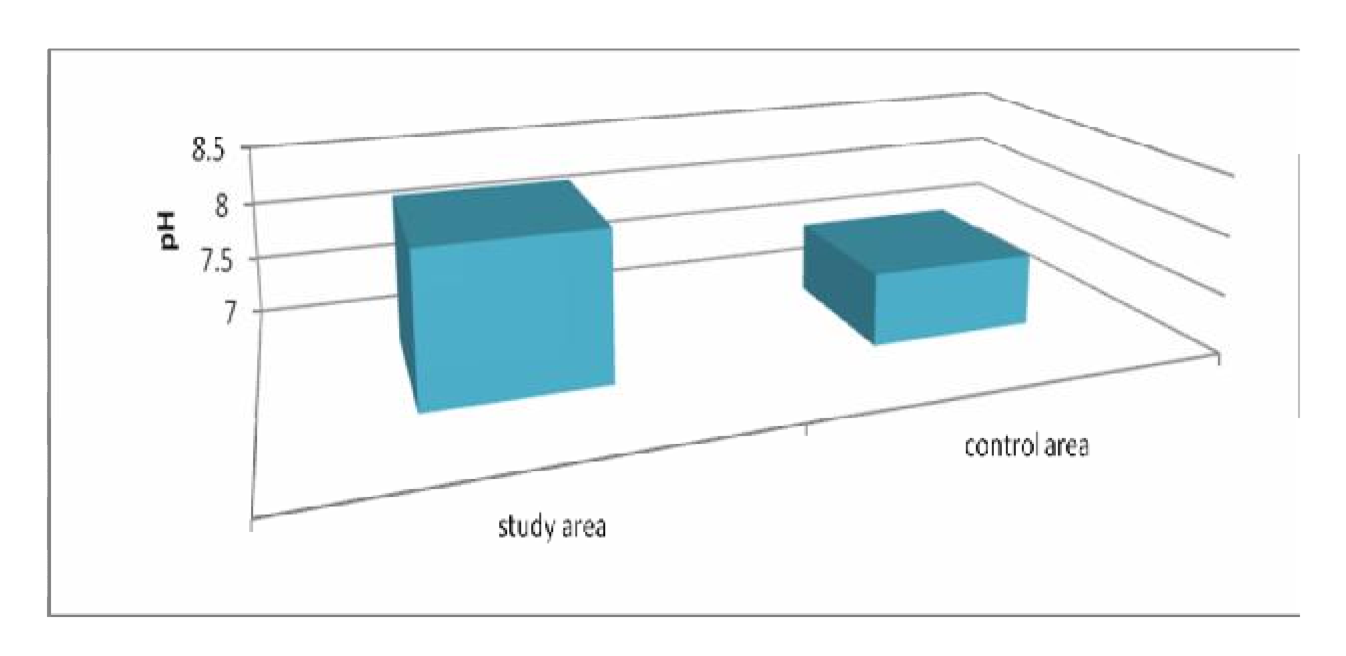


Figure (3.1)
Average pH values for study area and control area

3.1.2. Electrical Conductivity

Electrical conductivity (EC) measurements obtained by saturated water extraction of soil samples are shown in Table (3.1). In general, the conductivities of the water extracts in the study area was ranged from 1.3 to 24.6 mS/cm with an average 9.94 mS/cm for all soil samples, which was higher than EC value in the control area (3.40 mS/cm). Figure 3.2 showed significant differences in EC values between study area and control area. This was due to increase in total dissolved salts in the study area. (Jaradat et al., 2005; Bedell et al., 2006)

Saline soils are those which have an electrical conductivity of the saturated soil extract of more than 2 dS/m at 25°C. So, according to the definition of Saline soils the result of EC shows that all soil samples of the study area and control area are classified as non-saline soils. (Kristie, 2007)

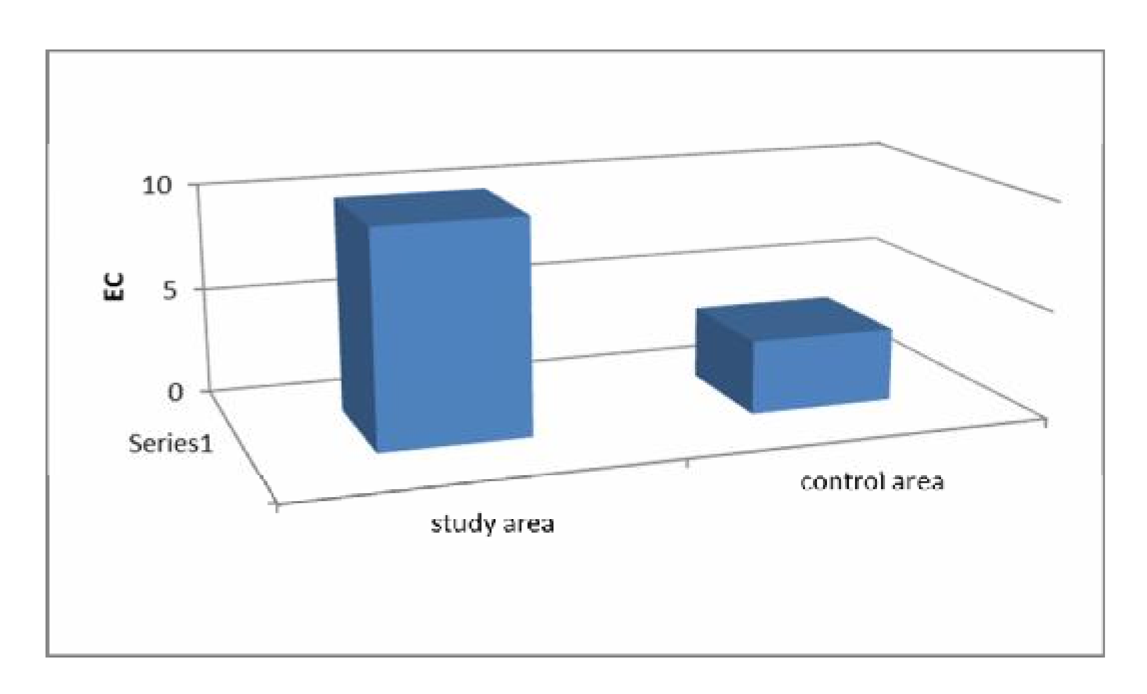


Figure (3.2)
Comparison of average EC (mS cm^{-1}) values for study and control area

3.1.3. Total organic carbon (TOC %)

The total organic carbon was determined according to Walkley-Black´s wet oxidation method and the results are presented in Table 3.1. However, the TOC% in the study area was ranged from 0.91 to 3.26% with an average 2.19% for all soil samples, which is higher than the TOC in control area (0.85%), as shown in figure 3.3. The significant differences in TOC% values between study area and control area can be attributed to the organic contaminants caused by kitchen residue, animal residue, cartons, paper, and car emissions such as fuel and oils. The maximum TOC was found at sites 37 and 60 (3.26) and the lowest at site 23 (0.67%).

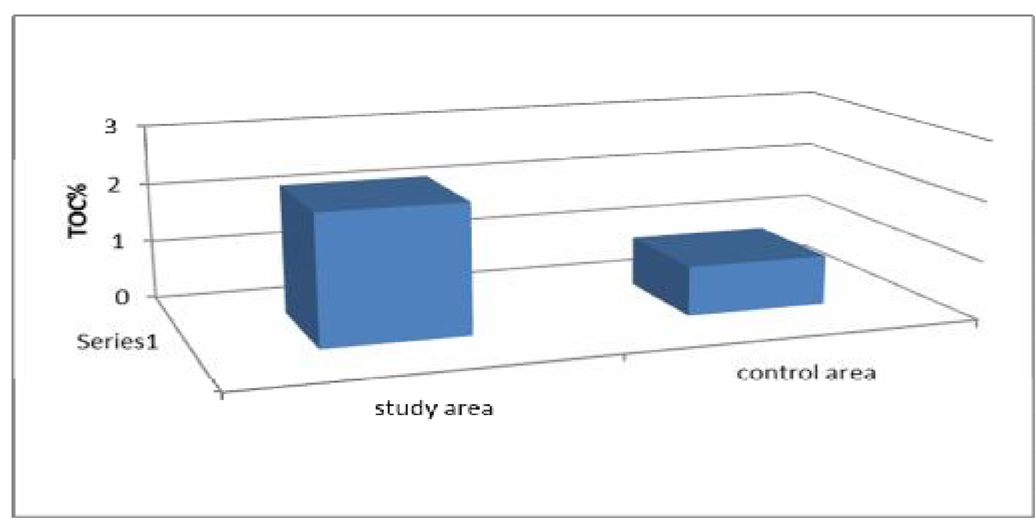


Figure (3.3)
Average TOC% values for study area and control area

3.1.4. Calcium Carbonate Content ($CaCO_3$%)

Percent of $CaCO_3$ was determined for eight samples namely: 1, 10, 20, 30, 40, 50, 60, and 70. The results are presented in table 3.2. The $CaCO_3$ among all selected samples ranged from 27.20% to 61.67% which located within the average value in Jordanian soil.

Table (3.2)
Calcium carbonate content $CaCO_3$ %

Sample	Calcium Carbonate content $CaCO_3$ %
1	36.99
10	51.28
20	51.18
30	61.67
40	51.84
50	60.81
60	27.20
70	37.15

The correlation between physical and chemical soil parameters (pH, EC,$CaCO_3$ and TOC) of soil samples was determined in the study area, Table 3.3. There were no significant correlation between pH and EC (r= -0.25). A weak correlation was found between TOC% and EC value (r= 0.19) followed by pH value and TOC% (r= 0.02). These results showed that the pH, EC and TOC values were independent on each other.

Table (3.3)
Correlation matrix between physical and chemical soil parameters

	pH	**EC**	**TOC%**	**$CaCO_3$%**
pH	1.00			
EC	-0.25	1.00		
TOC%	0.02	0.19	1.00	
$CaCO_3$%	0.34	-0.41	-0.42	1.00

3.1.5. Soil texture analysis

Soil texture was determined for eight soil samples namely: 1, 10, 20, 30, 40, 50, 60, and 70 using the hydrometer method. The results are presented in table 3.4. The soil texture among all selected samples was ranged from sandy loam to loamy sand. The average of these percentages were calculated and cited on the US Department of Agriculture (USDA) soil texture triangle to determine the soil texture, which is shown in figure 3.4.Trace metals have been shown to become mobile in soils through association with the dissolved organic matter , especially in sandy soils and soils with a high pH(Ge et al., 2000). The sand was the dominant soil fraction among the clay and silt fractions and composted of more than 80% of the soil texture.

Table (3.4)
Soil texture for selected samples.

sample	Slit%	Clay%	Sand%	Texture
1	8	12	80	Sandy loam
10	6	12	82	Sandy loam
20	8	16	76	Sandy loam
30	8	8	84	Loamy sand
40	10	10	80	Sandy loam
50	10	8	82	Loamy sand
60	8	10	82	Loamy sand
70	8	12	80	Sandy loam
average	8.25	11.00	80.75	Loamy sand

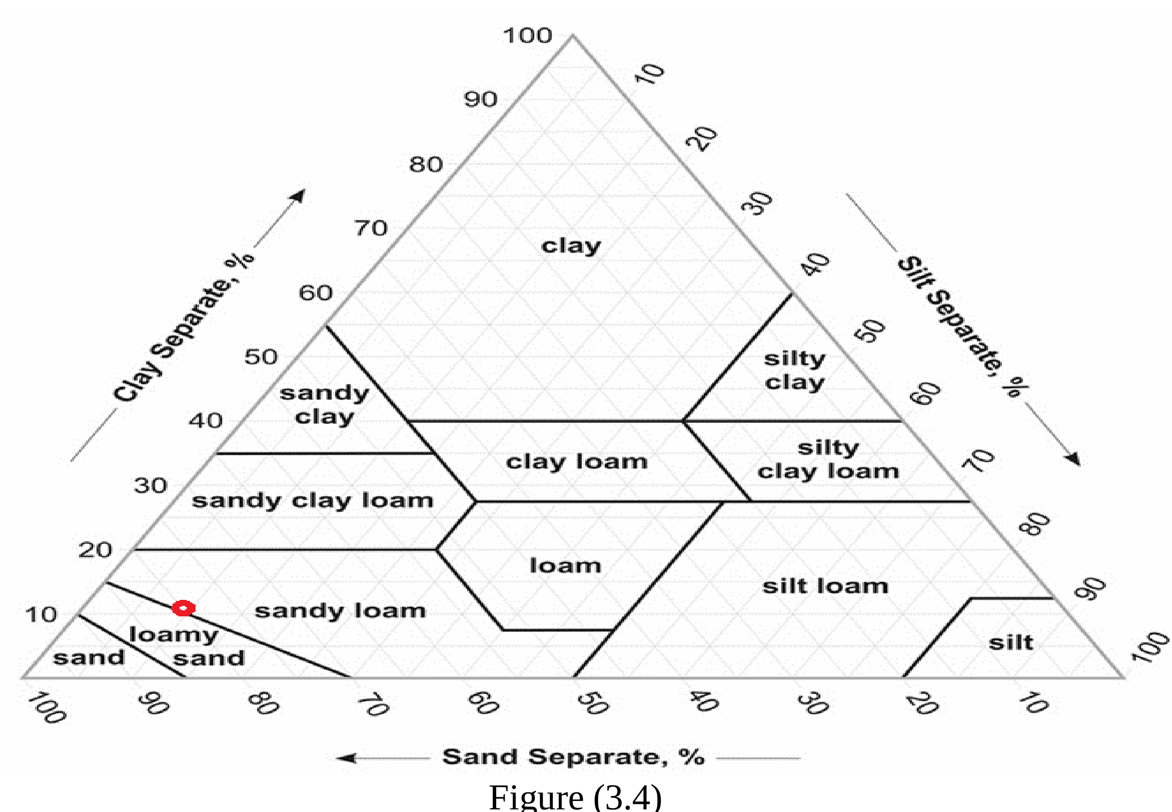


Figure (3.4)
Soil texture triangles according to USDA soil.

3.2. Analytical Method Validation

Eight metals (Zn, Ni, Pb, Cu, Co, Cr, Fe, and Mn) were analyzed quantitatively based on external calibration curves for series of standard solutions using flame atomic absorption spectrometer (FAAS). The calibration curves for these elements were determined with best regression lines with correlation coefficient (r^2>0.995), as shown in figure 3.5. The blank solutions showed no appreciable values of heavy metals and their values were ignored. For the degree of accuracy for total heavy metal digestion, a certified reference material (CRM) was digested using the same analytical procedure used for soil samples and analyzed subsequently using FAAS. The results were compared with certified CRM values and the percent recovery was computed. It was found that the procedure B which composed of a mixture of HNO_3, HCl and HF using the microwave had a robustness procedure with recovery ranging from 78.66% to 119.11% with relative standard deviation (RSD%) of less than 10%.

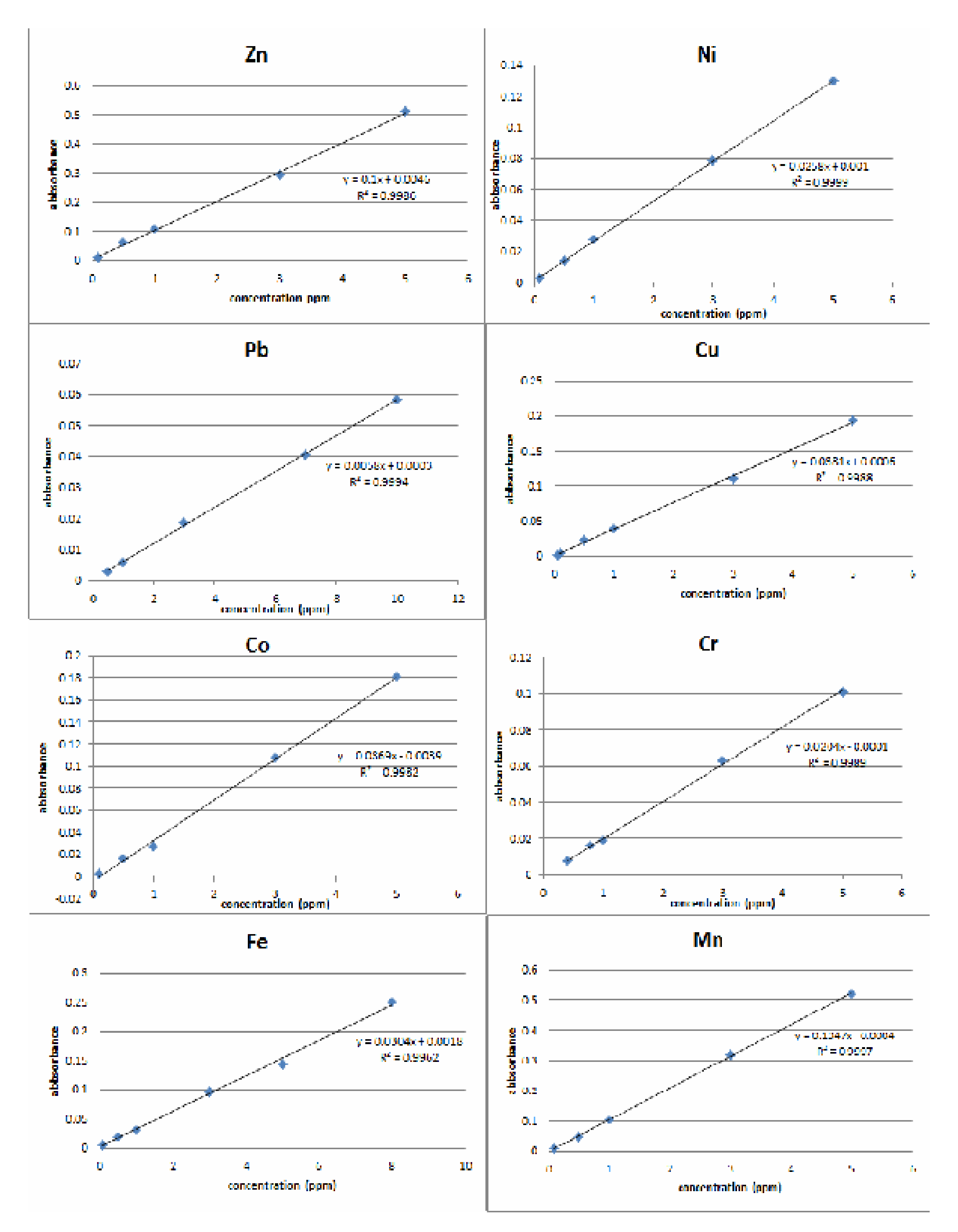


Figure (3.5)
Calibration curves for the heavy metals.

The LOD and LOQ were determined based on the statistical formula in sections **2.5.2.1** and **2.5.2.2** which was calculated for blank samples (6 replicates) after performing the complete digestion and analysis procedure in similar manner as the soil samples (Christian, 2004). The results of validation parameters including recovery rates, standard deviation of recovery rate and its relative standard deviation, in addition to the limit of quantitation and limit of detection for all heavy metals on basis of blank samples are presented in table 3.5.

Table (3.5)
Analysis of heavy metals (mg/kg) for CRM soil (MAPEP-07-MaS18), recovery (n=3), LOD (n=6).

Element	Grand mean	Measured Value	Recovery %	±SD	RSD	LOD*	LOQ**
Zn	284.0	223.8	78.66	2.99	1.29	0.120	0.362
Ni	129.0	147.2	114.14	2.01	1.37	0.066	0.200
Pb	77.0	83.6	108.62	3.02	3.61	0.083	0.250
Cu	85.40	76.2	89.23	6.03	7.91	0.120	0.362
Co	38.7	46.1	119.11	2.02	4.34	0.106	0.321
Cr	63.9	61.3	95.95	6.04	9.85	0.120	0.362
Fe	__	__	__			0.123	0.371
Mn	__	__	__			0.069	0.210

*LOD=3.3(s/S), determined for blank (n=6) according to the digestion and analysis method for each element using the element calibration curves.
**LOQ=10(s/S)

3.3 Heavy Metals Content

The heavy metals concentration (mg kg^{-1}) in all investigated soil samples are illustrated in Table 3.6. The variation in the concentration of heavy metals within different sites may be due to differences in their sources and the predominant soil physicochemical conditions and complex reactions such as adsorption, precipitation and redox conditions that may took place. The following sections are presenting the data of each individual element using the Geographic Information System (GIS) mapping technique (ArcView 3.3) after inserting the sampling sites coordination (latitude and longitude) to the Google earth map and the concentration of each element among all sampling sites.

Table (3.6)
Concentration of heavy metals (mg/kg) in soil samples of study area

Sample #	Zn	Ni	Pb	Cu	Co	Cr	Fe	Mn
1	82.05	137.65	102.49	38.87	97.88	68.42	881.36	124.12
2	76.67	157.35	103.10	31.83	90.82	82.44	1757.19	126.69
3	70.18	182.91	123.00	32.19	94.89	84.84	1945.46	133.69
4	62.2	184.60	131.13	37.30	92.16	61.32	1247.62	131.39
5	73.13	204.69	134.09	41.67	94.61	52.97	1301.62	128.92
6	70.46	219.73	149.46	44.60	97.66	70.90	1442.68	139.72
7	86.19	210.43	159.50	53.83	99.77	77.44	1523.73	133.20
8	46.11	216.10	155.95	42.26	86.87	59.67	1053.11	121.18
9	54.54	228.00	166.81	45.47	92.87	70.25	1392.02	132.75
10	87.04	261.53	183.57	53.19	92.12	76.50	1870.20	151.33
11	76.05	252.85	183.70	60.23	92.18	62.95	1679.72	145.60
12	62.79	236.48	194.34	55.23	94.65	58.58	1344.65	141.04
13	61.14	257.36	208.71	61.27	87.97	72.35	1448.32	140.98
14	51.90	285.68	232.97	58.56	93.26	55.26	831.85	136.29
15	69.58	290.66	232.75	58.65	91.55	85.26	1896.39	147.84
16	78.58	327.76	243.31	65.23	90.42	89.84	1869.36	170.90
17	55.09	296.51	247.58	61.22	100.03	83.20	1562.66	162.70
18	87.93	160.68	250.70	68.30	101.57	90.44	2235.86	162.14
19	74.40	158.18	274.33	70.32	104.01	85.80	1830.53	163.29
20	95.59	173.06	279.51	75.28	99.48	98.85	2317.37	182.95
21	66.69	166.09	283.49	75.26	91.85	67.24	1455.93	169.80
22	55.33	166.51	293.85	62.56	89.53	55.31	962.72	169.35
23	58.54	200.18	335.15	80.04	90.42	81.43	1920.69	178.52
24	58.56	183.02	369.35	64.32	87.97	58.81	1121.58	175.87
25	91.32	213.17	55.88	74.17	97.37	84.28	1236.29	207.88
26	45.51	199.85	58.09	69.03	93.58	56.81	871.89	200.74
27	91.21	227.62	58.44	89.58	99.42	107.72	2159.05	219.48
28	83.54	224.67	58.44	83.89	99.42	78.55	1572.01	208.53
29	77.88	247.03	65.87	84.29	104.17	97.97	2152.61	230.73
30	88.99	255.96	70.70	85.44	100.13	90.67	1668.26	232.08
31	94.07	253.59	67.13	88.79	99.00	113.39	2289.07	239.48
32	92.41	265.94	61.04	86.25	102.00	141.08	2088.77	237.86
33	93.15	254.52	65.67	94.06	96.23	186.20	1303.26	228.59
34	82.64	280.77	68.38	100.95	104.67	159.59	1568.21	241.4
35	75.76	287.96	60.82	92.92	98.30	151.57	1704.71	239.91
36	88.42	294.25	73.47	101.12	104.85	254.03	1822.27	94.35
37	79.24	271.10	72.50	100.64	103.94	259.66	2228.48	100.33
38	113.88	297.29	73.20	108.61	99.97	234.34	2479.59	100.55
39	67.71	291.41	60.68	102.28	96.16	137.66	1048.56	91.26
40	71.17	318.14	70.77	109.54	99.77	181.40	1571.83	102.81
41	79.17	29.94	60.68	105.76	110.45	325.56	2016.4	111.63

Continue Table 3.6

42	97.35	30.26	77.72	111.28	99.37	220.02	2007.08	120.93
43	57.5	41.13	68.52	98.95	102.97	347.00	1288.24	106.41
44	85.49	41.06	78.36	114.55	103.27	228.40	1929.66	121.24
45	58.39	17.42	70.82	107.00	103.65	217.80	1586.22	110.55
46	86.81	30.07	72.50	114.90	95.43	439.73	2157.72	126.68
47	57.32	45.54	70.37	115.00	103.94	307.22	1504.20	119.64
48	76.62	49.28	68.33	117.05	97.92	250.20	1936.32	127.70
49	95.68	71.80	70.91	118.07	104.27	325.15	1976.14	143.59
50	90.61	82.84	73.77	126.15	96.13	416.76	1571.37	134.39
51	79.60	81.91	63.05	112.12	96.48	370.15	1613.46	135.80
52	79.41	91.22	65.46	125.03	111.58	518.57	2268.02	157.79
53	58.66	94.14	67.50	116.92	95.31	226.03	1672.44	145.62
54	86.24	106.89	73.15	121.61	107.73	216.72	2204.72	155.86
55	80.15	116.38	63.58	124.89	107.84	338.14	1967.68	150.11
56	77.54	86.70	65.59	120.93	107.52	327.94	1443.09	153.01
57	83.83	112.57	75.90	134.23	104.27	380.29	1659.98	166.64
58	105.79	146.78	78.36	137.72	107.59	339.96	2419.50	169.50
59	111.10	160.80	90.70	136.28	112.25	450.40	2387.49	176.26
60	449.82	160.80	83.25	162.06	106.52	214.57	1818.02	193.78
61	589.65	179.97	87.78	169.08	112.17	337.28	1960.54	226.97
62	117.55	160.23	85.11	134.41	99.11	432.97	2020.69	170.27
63	278.10	150.75	77.97	150.09	108.00	495.72	1768.03	190.45
64	191.04	168.00	87.06	213.85	180.90	214.29	1726.33	178.98
65	72.40	180.90	74.36	129.84	109.19	462.20	1536.50	179.37
66	145.60	163.06	88.16	163.27	111.23	465.67	2039.89	180.88
67	104.00	212.13	80.77	145.90	110.35	386.27	2625.02	191.96
68	72.15	218.08	72.33	133.19	106.53	403.55	1736.61	185.82
69	82.00	220.25	72.86	130.68	104.46	234.54	1618.49	179.20
70	71.29	188.71	75.40	127.29	106.43	533.74	1954.50	182.96
71	89.56	222.46	70.53	145.75	114.66	386.00	2151.87	192.64
72	98.59	209.88	75.62	144.68	108.18	367.91	2676.02	200.62
73	130.77	196.53	79.23	141.84	105.44	339.52	2046.5	202.65
74	116.18	253.51	85.17	181.89	100.60	299.39	2658.79	215.70
75	198.45	212.04	100.87	147.54	105.33	292.35	1966.64	197.88
average	98.03	185.00	116.42	99.85	101.47	215.96	1767.62	163.33
control 1	67.17	91.39	94.50	17.55	26.90	57.27	630.94	94.95
control 2	58.61	90.66	96.92	15.30	29.77	49.24	534.99	126.25
control 3	28.84	89.89	91.02	24.11	23.37	37.92	597.77	44.34
control 4	64.47	94.29	86.26	17.09	25.64	54.25	627.04	118.11
control 5	52.07	89.85	113.37	19.96	28.33	48.21	684.73	96.36
control 6	43.26	91.13	100.10	20.91	31.42	43.26	650.60	70.35
average	52.40	91.20	97.03	19.15	27.57	48.36	621.01	91.73

3.3.1 Zinc

The average concentration of Zn in control samples was 52.40 mg/kg, while in the study area was 98.03mg/kg with a range between 45.51 and 589.65 mg/kg (Table 3-6). This indicating that the soil samples in the study area has higher level of Zn by two folds than the control area. In addition, as illustrated in Figure 3.4, the site 61 was found to have the highest Zn concentration followed by 60 and 64, respectively. Generally Zinc occurs naturally in soil (about 70mg kg^{-1} in crustal rocks) (Davies et al., 1988). The likely source of Zn in the study area is the foodstuffs that may contain certain concentrations of Zn as the waste dumped in the study area contained 52% kitchen residue from the daily income of solid waste (SWDF,2011; Wuana et al., 2011). Additionally, it was found that 94.67% of the investigated samples were contained concentration level below the range defined by (McGrath et al., 2001). The typical range of trace elements in non-polluted soil is illustrated in Table 3.7. The average Zn content was found in range 10-200 mg/kg for non-polluted soil (McGrath et al., 2001). However, only around 5% of investigated soil samples showed concentrations higher than this average, as shown in figure 3.4. The GIS contour map shows the concentration levels of Zn using the colored map index which was given in five ranges (50-150, 151-250, 251-350, 351-450, and 451-600 mg/kg), (Figure 3.6.)

Table (3.7)

Typical range of trace elements in non-polluted soil (McGrath et al., 2001).

Element	Range (mg/kg) (Total content)	Investigated area (mg/kg)
Zn	10 – 200	45.5 - 589.7
Ni	0.5 – 100	17.4 – 327.8
Pb	2.1 - 80	55.9 – 369.4
Cu	2 – 100	31.8 – 213.9
Co	1 – 25	86.9 – 180.9
Cr	5 – 250	52.9 – 533.7
Fe	-------	831.9 – 2676.0
Mn	20 – 3000	91.3 – 241.4

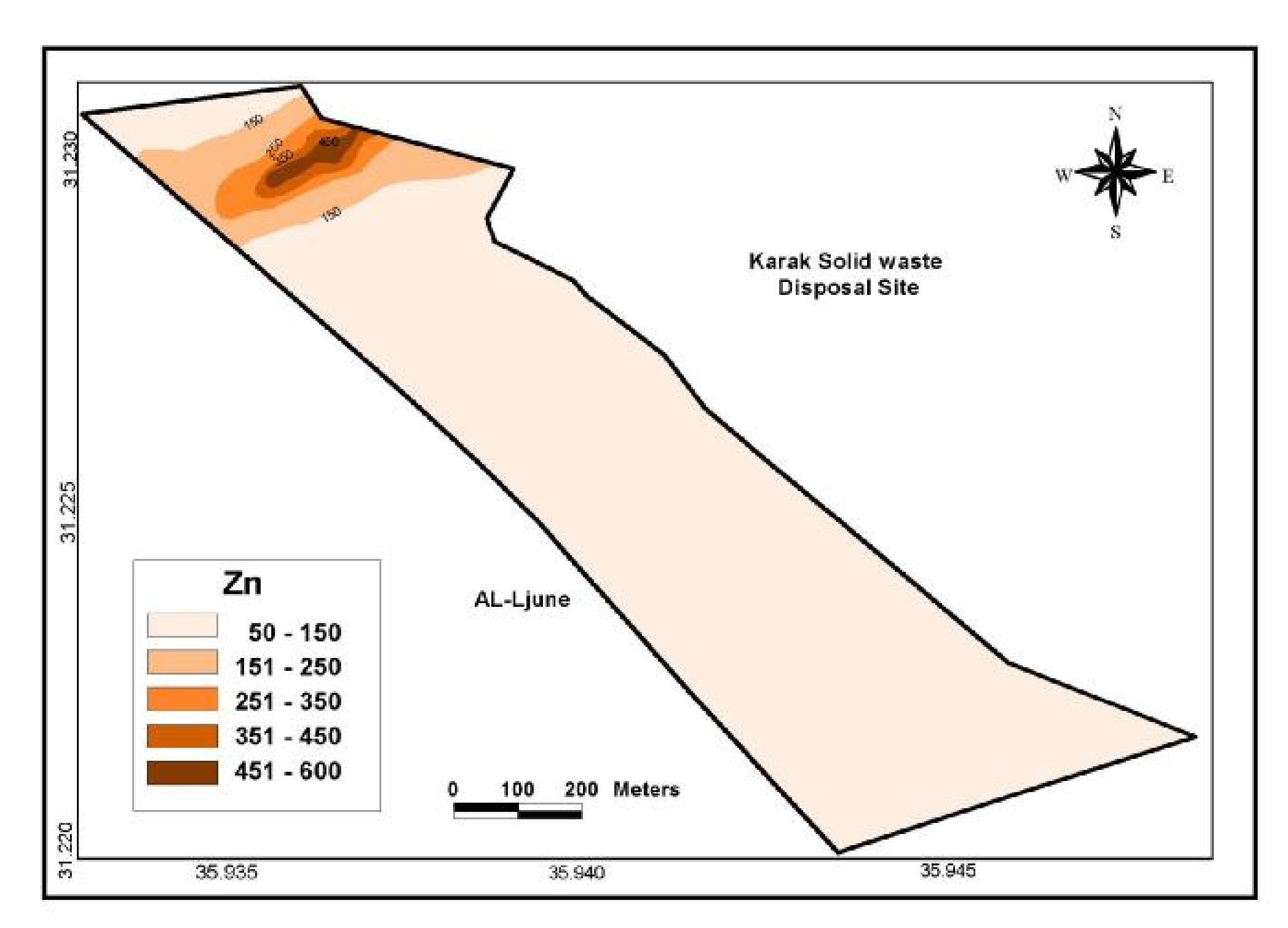


Figure (3.6)
Distribution of Zn concentrations in study area using GIS maps.

3.3.2 Nickel

The mean concentration of Ni in the control area was 91.20 mg/kg and in the study area 185.00 mg/kg with range between 17.42 mg/kg and 327.76 mg/kg. The samples of the study area have elevated level of Ni compared with that for the control area by 2 times. Different site have a high level of Ni but site 16 have the highest concentration (327.76 mg/kg) of Ni in study area, while site 45 showed the lowest concentration (17.42 mg/kg). It was found that 81% of the investigated samples contained concentration level above the Ni range given for non-polluted soil, as shown in table 3.7. The distribution pattern of Ni showed elevated environmental concentration among the whole sampling sites.

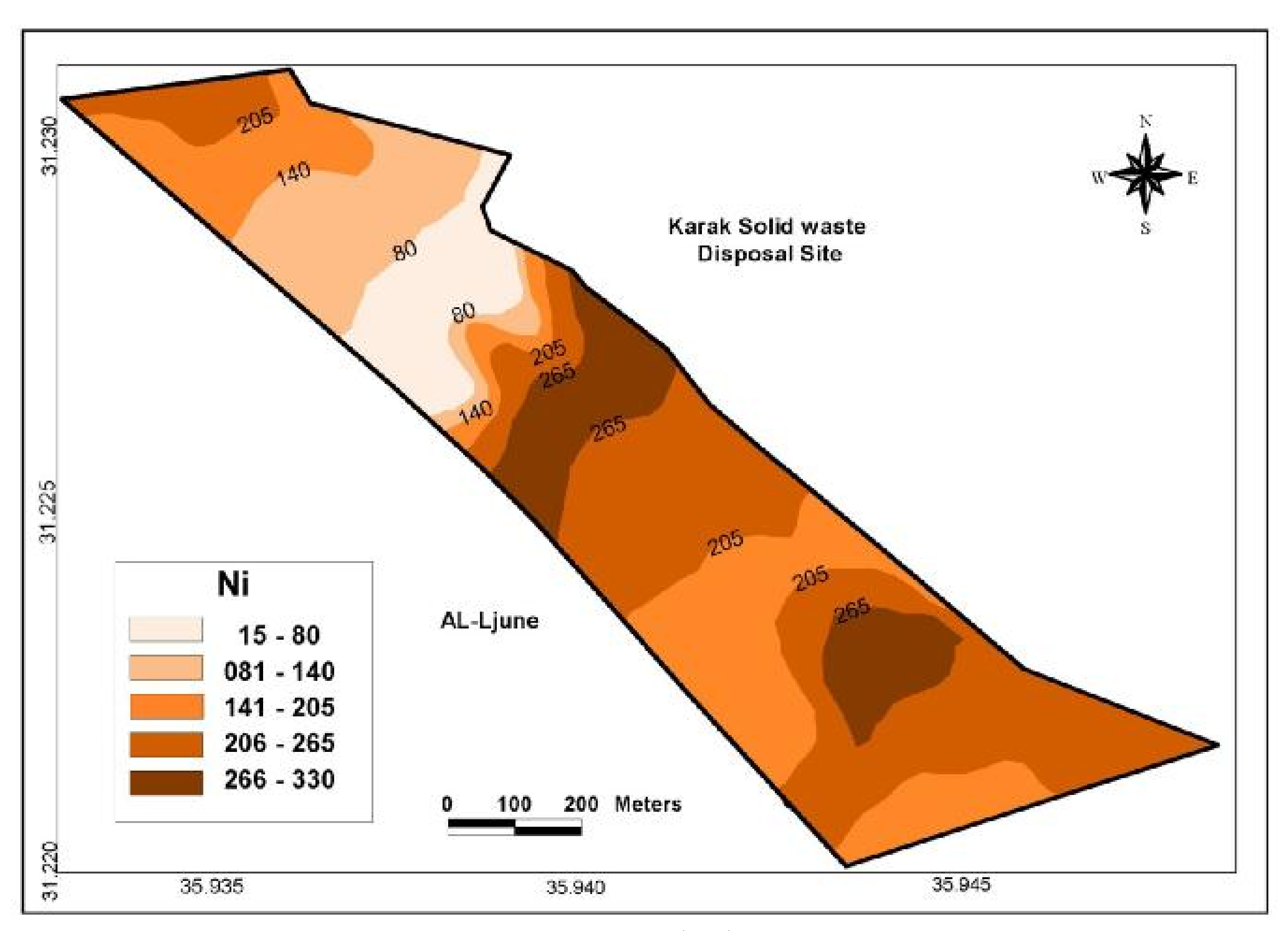


Figure (3.7)
The distribution of **Ni** concentration in study area using GIS map.

Nickel is used in many specific and recognizable consumer products, including stainless steel, alnico magnets, coinage, rechargeable batteries, electric guitar strings, microphone capsules, and special alloys. It is also used for plating and as a green tint in glass (Davis, 2000). The source of Ni might be due to the metals waste that consisted of 4% of the input waste at the investigated site.

3.3.3 Lead

Lead is considered one of the most important heavy metal that contributes to the contamination in environment. Its mean concentration in the control area was 97.03 mg/kg, while in the study area 116.42 mg/kg with a range from 55.88 mg/kg to 369.35 mg/kg. The results showed elevated concentration levels of Pb than the control area. It was found that 57.33% of

investigated samples contained concentration levels within the range of trace element of non-polluted soil (Table 3.7). While 42.67% of the samples contained concentration levels above the range.

The main source of Pb in the study area might be attributed due to the automobile exhaust (Jaradat et al 1999). Lead found in fuel as tetraethyl lead and used as anti-knock agent in the combustion of gasoline (Divrikli et al., 2003). The distribution pattern of Pb showed elevated environmental concentration in the southern part of the investigated area among the whole sampling sites, as shown in figure 3.8. Which is located close to the entry site of transportation vehicles to the site. The main entrance location of the solid waste disposal site is shown in figure 2.1, where all vehicles transporting waste or visiting the site are passing this road only.

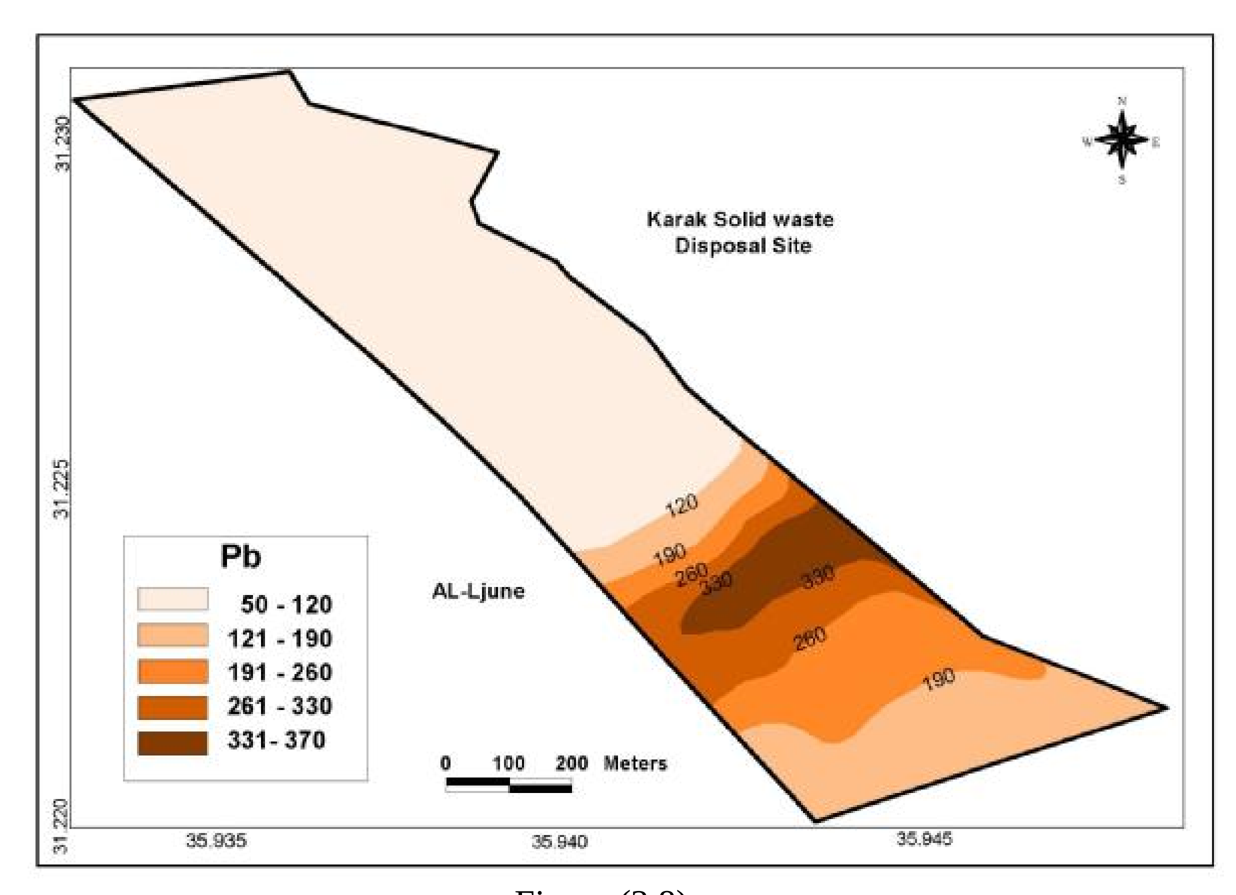


Figure (3.8)
The distribution of **Pb** concentration in study area using GIS maps.

3.3.4 Copper

The average concentration of Cu in the study area was 99.85 mg/kg and it is almost five time higher than that of the control area 19.15 mg/kg, as shown in table 3.5. In addition as illustrated in figure 3.7 the highest concentration of Cu was found in the sites 64 and 74, while, the lowest Cu concentration was found in the sites 2 and 3. It was found that 49.33% of the investigated samples contained concentration level within the range of Cu of non-polluted soil, (Table 3.7). While 50.67% of the sample contains concentration level above the range. The variation of Cu level between sampling sites may be due to unspecific sources as wires of copper, electric device tailing, and plating materials using to protect the surface of the brass alloy itself (Kartal et al,2006). The distribution pattern of Cu in the investigated area showed that elevated environmental concentration dominate in the north-western part of the disposal site, as shown in figure 3.9.

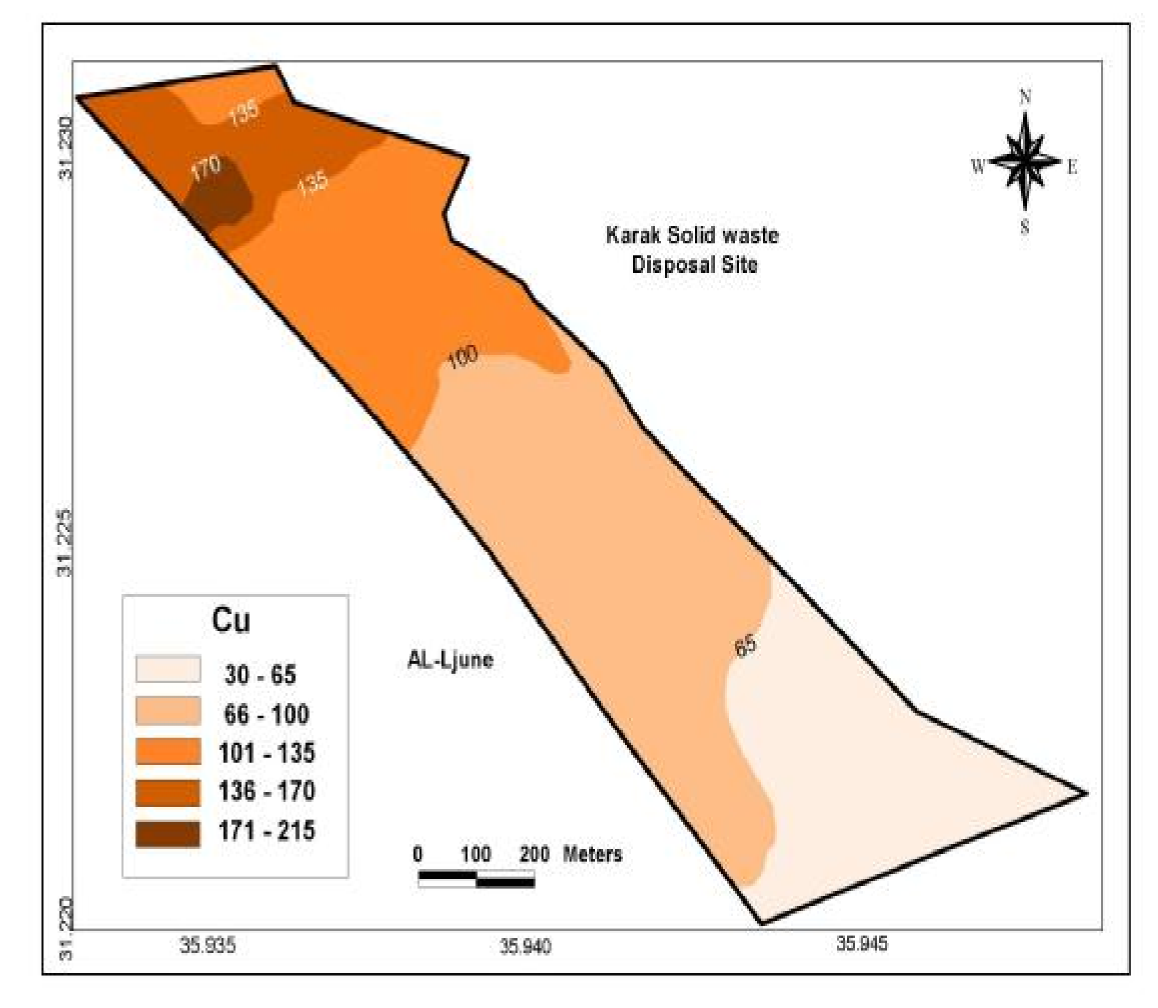


Figure (3.9)
The distribution of **Cu** concentration in study area using GIS maps.

3.3.5 Cobalt

The mean concentration of Co in the control area was 27.57 mg/kg and in the study area was 101.47 mg/kg with a range between 86.87 mg/kg and 180.9 mg/kg. The study area has elevated concentration level of Co compared with that for the control area about four times. Site 64 has the highest concentration of Co in the study area and site 8 the lowest, (Table 3.7). The average concentration of Co in the non-polluted soil range 1-25 mg/kg, therefore, the investigated area can be classified as contaminated soil according to (McGrath et al., 2001). The potential sources of Co might be due to the plastic waste that is 5% of the input waste. (Ross 1996).

The distribution pattern of Co in the investigated area showed that elevated environmental concentration dominate in the whole site and particularly highest concentrations were found in the north-western part of the disposal site, Figure 3.10.

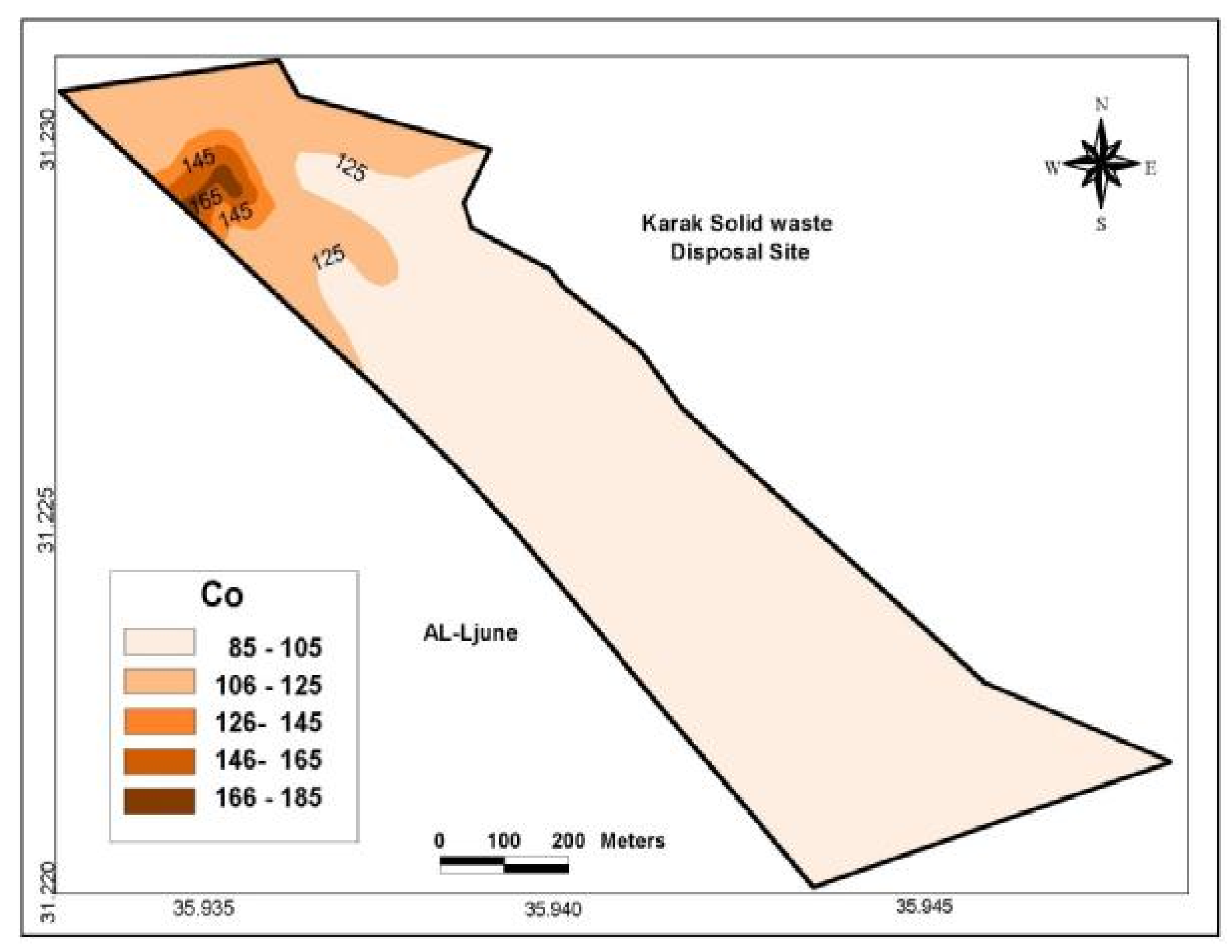


Figure (3.10)

Distribution of **Co** concentrations in study area using GIS maps.

3.3.6 Chromium

The average level of Cr in the study area was 215.96 mg/kg which is almost 4.5 times higher than that of the control area of 48.36 mg/kg, (Table 3.6). In addition as illustrated in figure 3.9 the highest concentration of Cr was found in sites 70 and 52, while, the lowest Cr concentration was found in the sites 5 and 26. It was found that 62.66% of the investigated sample contained concentration level within the range of trace element of non-polluted soil, Table 3.7. While 37.34% of the samples contained concentration levels above the range (McGrath et al., 2001). Major sources of Cr contamination might include releases from electroplating processes and the disposal of Cr-containing wastes (Smith et al., 1995). The distribution pattern of Cr in the investigated area showed that elevated environmental concentration dominate in the north-western part of the disposal site, Figure 3.11.

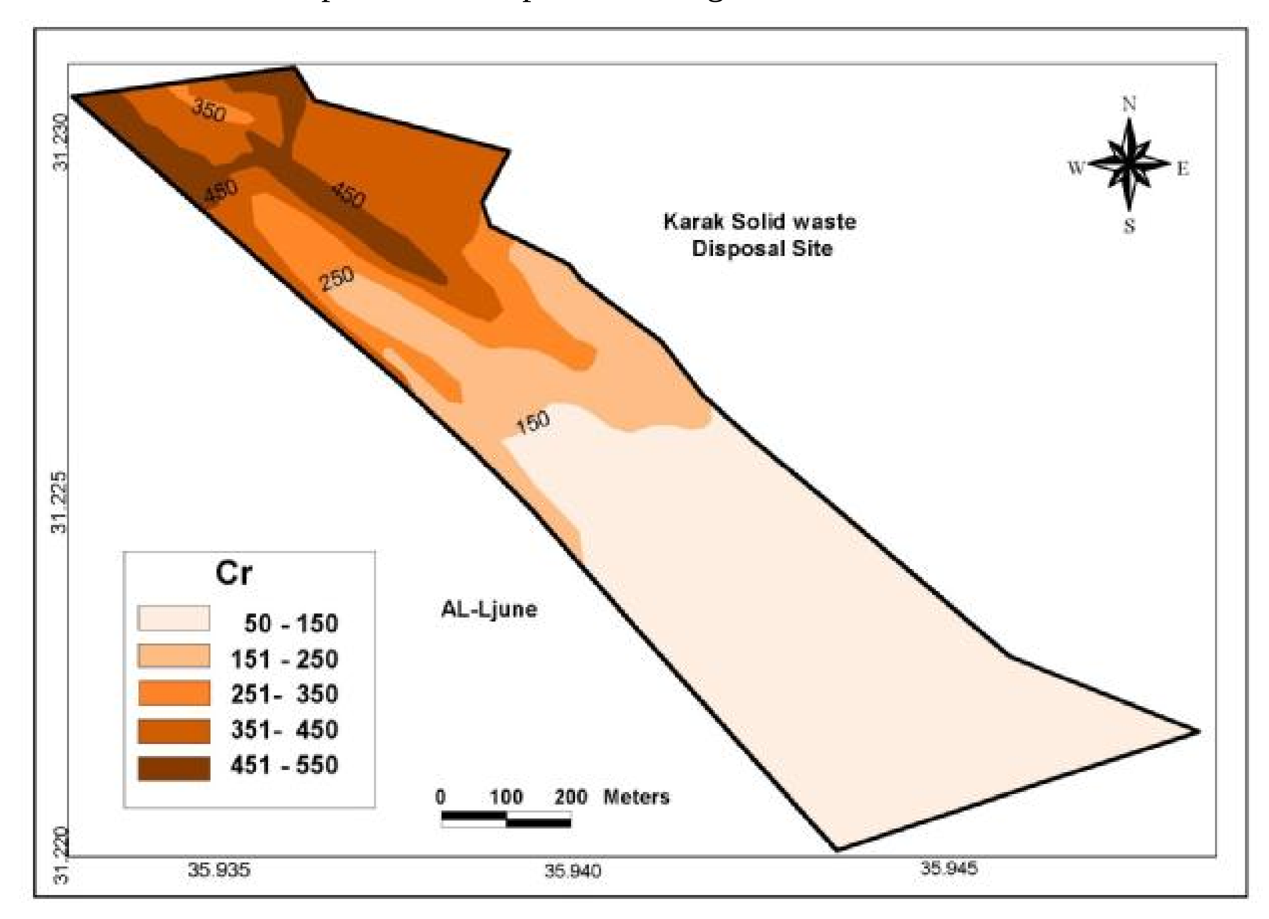


Figure (3.11)
Distribution of **Cr** concentrations in study area using GIS maps.

3.3.7 Iron

The mean concentration of Fe in the control area was 621.01 mg/kg and in the study area 1767.62 mg/kg with the range between 831.85 mg/kg and 2676.02 mg/kg. The samples of study area have elevated concentration levels of Fe compared with those for the control area by 3 times. Site 73 showed the highest concentration of Fe in the study area. This could be because the dumping site was enriched with iron through metals deposition. Besides, iron is one of the most abundant mineral in the soil (Brady 1982). The distribution pattern of Fe in the investigated area showed that elevated environmental concentration scattered overall the disposal site and the highest concentrations were dominated in the north-western part, Figure 3.12.

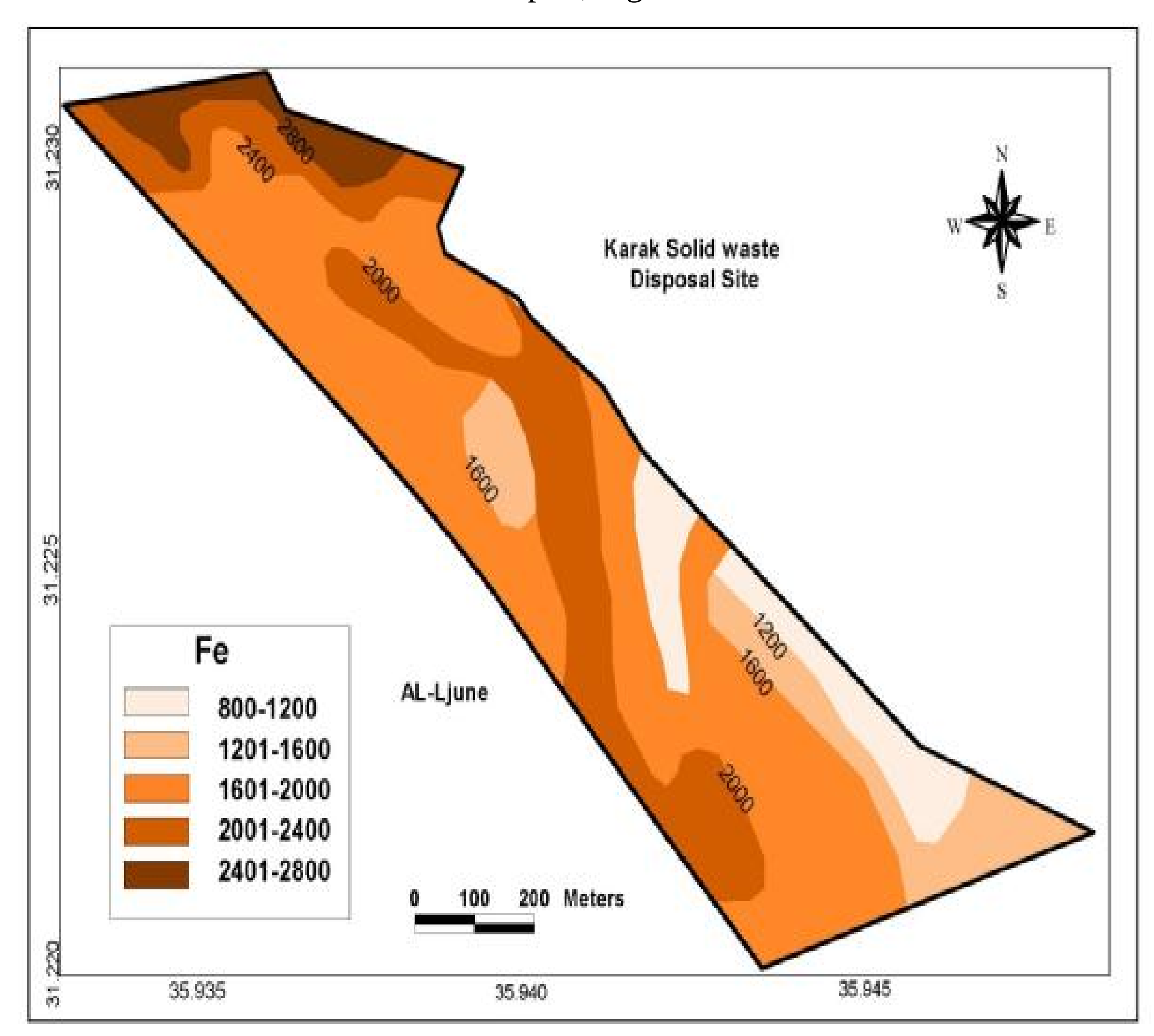


Figure (3.12)
Distribution of **Fe** concentrations in study area using GIS maps.

3.3.8 Manganese

The average concentration level of Mn in the control area was 91.73mg/kg which is lower than that in the study area (163.33 mg/kg), as shown in table 3.6. The highest concentration of Mn was found in site 34 and the lowest at site 39. The source of Mn in the study area may be due to enrichment with Mn through metals deposition. Besides, it is one of the most abundant mineral in soil (Brady, 1982). The distribution pattern of Mn in the investigated area showed that elevated environmental concentration scattered overall the disposal site in similar manner to Fe, while the highest concentrations were dominated in the central part of the disposal site, (Figure 3.13). The investigated area can be classified regarding Mn concentration as non-polluted soil according to (McGrath et al., 2001). Cr, Zn and Cu showed similar distribution pattern, while Fe, Mn and Ni had also similar distribution pattern which scattered on the whole investigated area.

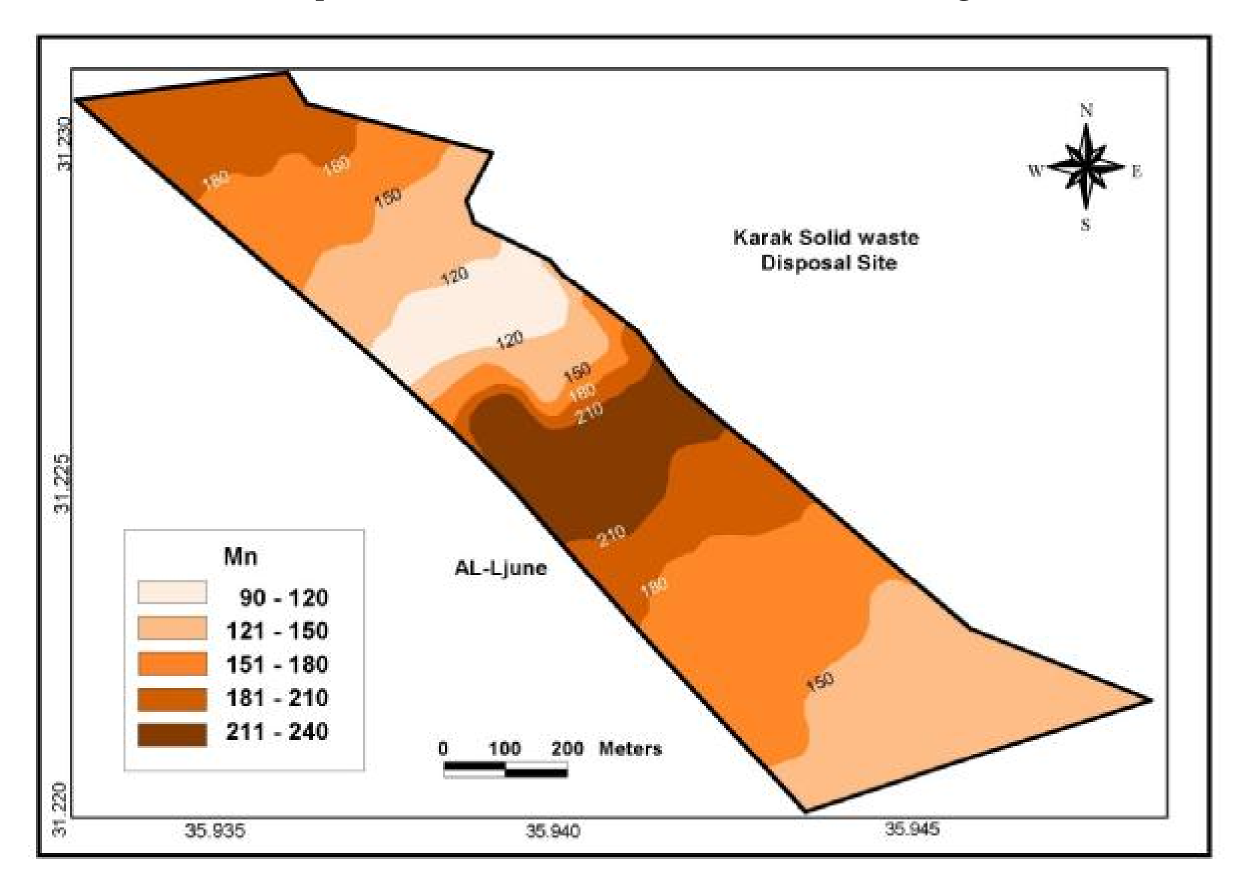


Figure (3.13)
Distribution of **Mn** concentrations in study area using GIS maps.

3.4 Correlations between heavy metals

Table 3.8 shows the correlation coefficients between the investigated heavy metals in the study area. Significant correlation has been found between Cu and Co (r=0.69), Cr (r=0.78), and Fe (r=0.5), which mean probably the same source of each pair of these metals. However low significant correlations were observed between Zn with Cu (r=0.49), Co with Cr (r=0.42), Cr with Fe (r=0.46); while there were insignificant correlation between other metals. This could mean independent source of those metals.

Table (3.8)

correlation matrices between heavy metals in study area

Element	Zn	Ni	Pb	Cu	Co	Cr	Fe	Mn
Zn	1							
Ni	-0.04	1						
Pb	-0.17	0.22	1					
Cu	0.49	-0.27	-0.51	1				
Co	0.33	-0.17	-0.36	0.69	1			
Cr	0.25	-0.43	-0.54	0.78	0.42	1		
Fe	0.21	-0.05	-0.23	0.50	0.30	0.46	1	
Mn	0.30	0.31	-0.08	0.27	0.17	0.03	0.24	1

3.5 Correlations between heavy metals and soil properties

Table 3.9 shows the correlation between heavy metals concentrations in the study area and the physical and chemical soil parameters. The result showed that there was strong significant correlation between the concentration of Pb and Texture (clay %), low significant correlation between the concentration of Cr and EC, while; there were insignificant correlation between other metals and soil parameters.

Table (3.9)

Correlation matrices between heavy metals concentration and physical and chemical soil parameters

Parameters	Zn	Ni	Pb	Cu	Co	Cr	Fe	Mn
pH	0.05	-0.08	0.14	-0.16	-0.09	-0.13	0.09	-0.10
EC	**0.27**	-0.08	-0.30	**0.38**	0.19	**0.49**	0.28	0.09
TOC%	**0.28**	-0.08	-0.19	0.21	0.09	0.24	0.22	-0.08
Texture (clay %)	**-0.14**	-0.03	0.84	-0.41	-0.01	-0.23	0.42	-0.03
$CaCO_3$%	-0.63	0.18	0.13	-0.27	-0.63	-0.11	0.12	-0.02

3.6 Enrichment Factors

Enrichment Factors (EF) of heavy metals in the study area were calculated to give an indication for the degree of pollution of the soil with

these metals in study area. Iron (Fe) was used as a reference element, assuming that its anthropogenic sources to the soil were negligible (Samara et al., 2005, Chang et al., 2005), due to the crust-dominated element (Kartal et al. 2006). The EF was computed in accordance with the formula reported elsewhere (Remann et al,2005; Samara et al. 2005; Massadeh,2003; Al-Momani., 2003; Bergamaschi et al., 2002; Chang et al., 2005). The EF calculation methodology was as follows:

$$EF = \frac{[C_x/C_{Fe}]}{[C_x/C_{Fe}]_{crust}}$$

Where:

$[C_x/C_{Fe}]$: the ratio of concentration of the metal being determined (C_x) to that of Fe (C_{Fe}) in the soil sample.

$[C_x/C_{Fe}]_{crust}$: the ratio of the concentration of metal X to that of Fe in the earth crust. Where the concentration of Fe in the earth crust is (56300 mg/kg)

(Taylor,1964) compilation for crusted abundance's was used in the calculation of Enrichment factor. Generally, EF values less than 5.0 are not considered significant, because such small enrichments may arise from differences in the composition of local soil material and reference soil used in EF calculations (Chang et al, 2005). The three contamination categories were recognized based on the enrichment Factor values as: less enriched (EF<10), intermediately enriched (10<EF<100) and highly enriched (EF>100).

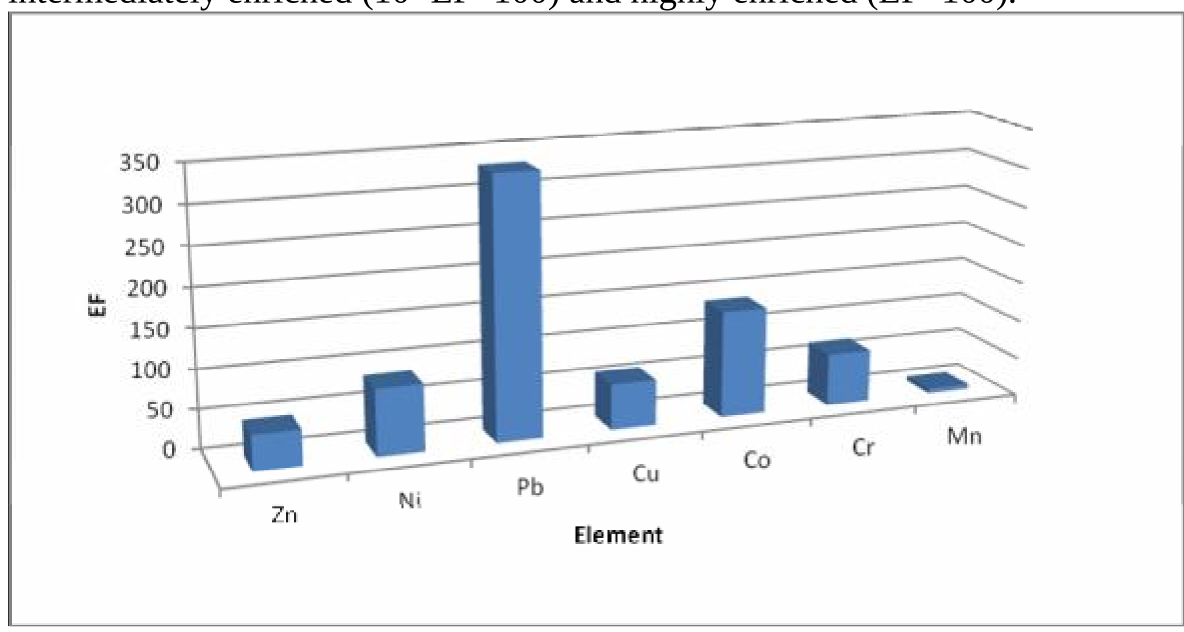


Figure (3.14)

Average enrichment factors for Heavy metals in study area

As shown in Figure 3-14, Mn had the lowest EF average value (5.78), which indicated that the background was the main source of Mn in the studied samples. While, Zn, Ni, Cu, and Cr have been intermediately enriched with EF 45.21, 85.11, 58.53, and 67.19, respectively. Finally, Pb and Co are highly enriched 332.57 and 137.03, respectively. The order for enrichment factor found as follows: Pb>>Co>Ni>Cr >Cu> Zn>Mn. The EF was greater than 5 for all metals in the investigated area which means that the anthropogenic activities as the main sources for these metals.

3.7 Bioavailable heavy metal concentrations

The determination of total heavy metal concentrations in soils might be useful to predict the potential environmental risk posed by these metals, but do not always provide a good indicator of metals labile fraction available for plant uptake and their potential mobility in the soil (Gasparatos et al, 2001). The severity of pollution depends not only on total heavy metal content of the soil, but also on the proportion of their mobile and bioavailable forms, which are generally controlled by the texture and other physicochemical properties of soils (Imperatoa et al., 2003).

Consequently, the availability of the element is very important when assessing the effect of soil contamination on plant metal uptake and related phytotoxic effects (Mench et al., 1994). The bioavailable concentrations of the heavy metals were determined in the following soil sample (1, 10, 20, 30, 40, 50, 60, and 70). The results are presented in Table 3.10.

Table (3.10)

Bioavailable heavy metals concentrations (mg/kg) in the studied soils.

sample	Zn	Ni	Pb	Cu	Co	Cr	Fe	Mn
1	1.210	2.586	1.342	2.025	0.678	1.521	3.689	7.554
10	1.385	2.622	1.458	0.876	0.545	1.684	4.923	10.94
20	0.228	2.695	0.827	0.834	0.387	0.973	4.906	3.758
30	1.520	2.617	1.624	0.366	0.360	1.823	4.742	1.904
40	2.022	2.676	2.379	0.451	0.809	2.322	4.774	9.870
50	0.929	2.583	1.211	0.118	0.545	1.235	4.889	7.953
60	25.72	2.745	7.832	0.366	1.339	1.721	22.94	38.84
70	0.581	2.666	0.783	0.471	0.492	1.007	4.166	6.679

The percentages of the total metals extracted with DTPA could be used as a good indicator of the quantity of metal available for plants and to reflect their comparative mobility (Papafilippaki et al., 2007). The bioavailable metal content designated as (Extractable -DTPA/Total %) was calculated as follows:

Extractable -DTPA/Total %= (bioavailable metal concentration/total metal concentration) X100%

The results of the Extractable -DTPA/Total % for the average concentration of the above eight samples are shown in Table 3.11.

Table(3.11)

Bioavailable content as a percentage of the total metal content (Extractable -DTPA/Total %)

Element	**Average of total concentration mg/kg**	**Average of bioavailable concentrations mg/kg**	**Extractable - DTPA/Total %**
Zn	98.03	4.2	4.28
Ni	185.00	2.65	1.43
Pb	116.42	2.18	1.87
Cu	99.85	0.69	0.69
Co	101.47	0.64	0.63
Cr	215.96	1.54	0.71
Fe	1767.62	6.88	0.39
Mn	163.33	10.94	6.70

According to (Ulrich et al, 1999) the metals are potential available for plant uptake, if the bioavailability percentage is above 10%.

The bioavailable content as a percentage of the total metal content was low for all investigated samples which indicating a low availability of the metals. The low availability of these metals could be due to the high retention capacity of the studied surface soils, due to their strongly calcareous nature and to high presence of free calcium carbonate (Gasparatos et al., 2001; Pichering., 1982). Also, the basic soil pH, due to the presence of free calcium carbonate, affects the bioavailability of the heavy metals, because high pH values increase the adsorptive capacity of soils (Papadopoulos et al., 1988). In addition the reduction of soil pH values increases the bioavailability and the mobility of the metals (Adriano., 1986). However, sample number 60 showed extremely high concentration of metals that affect the Extractable -DTPA/Total % value, individually Extractable -DTPA/Total % was calculated too, and it showed lower bioavailability for all metals too, except for Mn which was 20% that might be due to high relative abundance in the earth crust (Brady, 1982).

The relative availability and consequently the comparative mobility of the studied metals in the surface soils of the studied area followed the following order : Mn> Zn>Pb> Ni> Cr> Cu> Co> Fe. Organic matter plays a significant role in the availability and mobility of the heavy metals in soils too. The humified organic matter is involved in the formation of soluble complexes especially with Mn, which during organic mineralization, become

more available for the plants (Leita et al., 1999). This can explain the higher availability of Mn in the studied soils, in relation to the other metals. On the other hand Fe is a strongly retained element on the soil in according with the results of this study (Andreu et al., 1999).

3.8 Statistical analysis

In order to investigate the distribution pattern of individual heavy metal in the investigated sites the samples were grouped for statistical purpose into 15 groups distributed horizontally in the investigated area, each group consisted of 5 samples.

The "principle component analysis" (PCA) was done using SPSS Statistics version 17.0 and the results have been graphically displayed as loading plots, Figure 3.13. The Principle component analysis categorized the 15 main groups into five categories. Category 1 is consisting of groups (4, 6 and 7); Category 2 is consisting of groups (1, 2, 3 and 5); Category 3 is consisting of groups (12, 13, 14, and 15); whereas, groups (9, 10, and 11) were within the Category4 ; and finally group (8) was classified as a separate Category 5 which was characterized by similar concentration profiles.

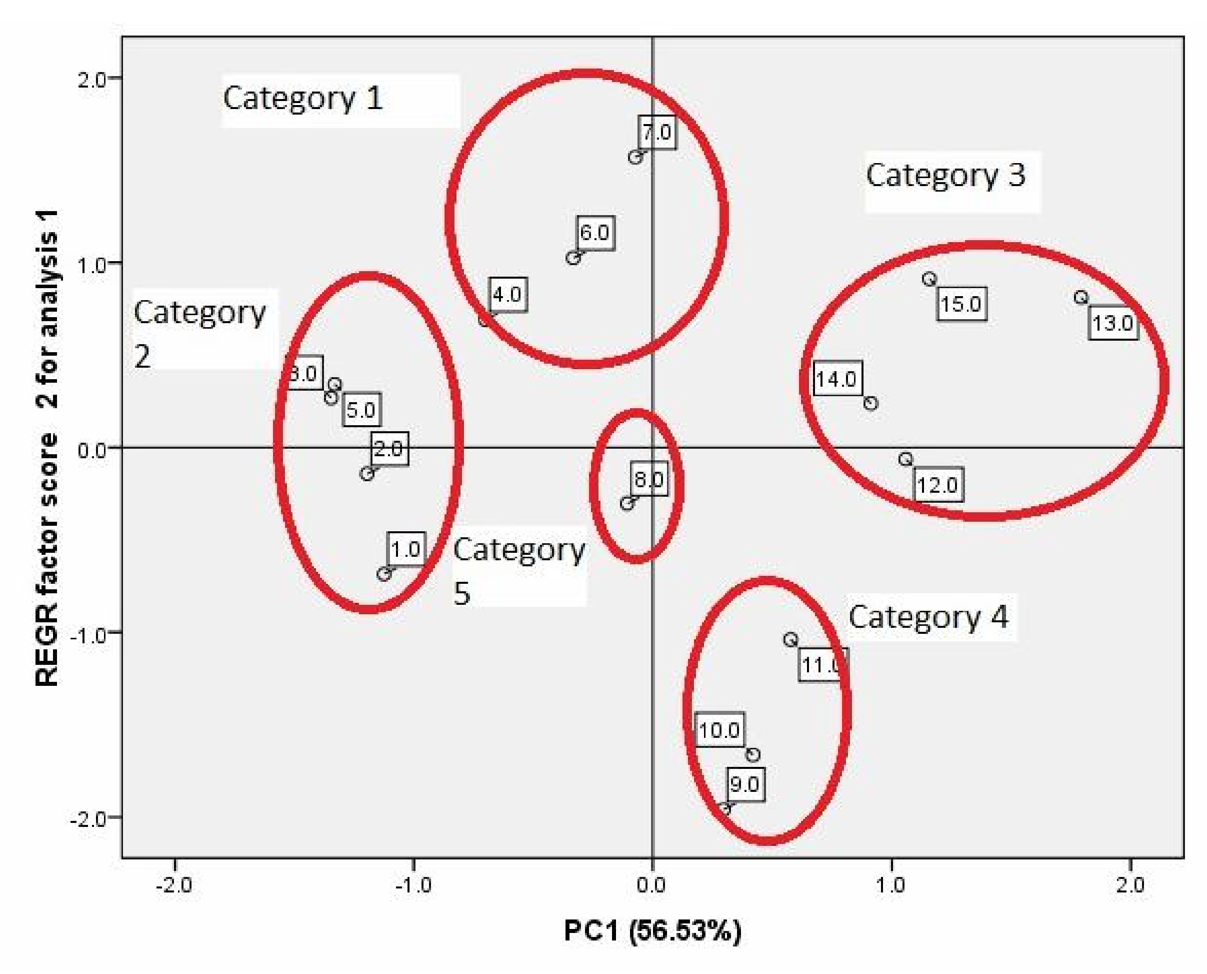


Figure (3.15)
Principle components analysis of 15 groups (1-15) based on concentration of heavy metals.

3.9 Conclusions and Recommendations

The following can be concluded from the current work:

1. Optimization and validation of the analytical method for trace metals using atomic absorption with microwave digestion was done using certified reference material and the validation parameters included the elements recovery rate, limit of determination (LOD), and limit of quantification (LOQ).
2. Looking for distribution pattern of trace metals in the surface soil in the investigated area using GIS mapping technique (Arc View 3.3).
3. Due to comparison of the elements concentration between the investigated area and reference control area. It was found that the investigated area is contaminated with different concentration levels of trace metals. According to the typical range of non-polluted soil it was found that the investigated

area had elevated concentration levels of Ni, Pb, Co and Cr more than the non-polluted.

4. The order for enrichment factor found as Pb>>Co>Ni>Cr >Cu> Zn>Mn. The EF was greater than 5 for all metals in the investigated area which can be explained due to the anthropogenic activities mainly waste dumping and vehicles which transporting the waste to the site as they were main sources for metals in the Karak Waste Disposal site.
5. The bioavailable content as a percentage of the available metals content to the total metal content was low for all investigated samples which indicating a low availability of the metals. The low availability of these metals could be due to the high retention capacity of the investigated soil which also affect the metals mobility.
6. It can be recommended that solid waste disposal site should be monitored for organic pollutants in soil and any generated leachate to evaluate the potential impact on soil and groundwater.
7. It can be also recommended an annual monitoring program of the disposal site after closing to prevent any future adverse impact on the surrounding environment. It can be also covered with a layer of clean soil and planted with different trees after stopping waste disposal to prevent direct contact with environmental conditions.

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